

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-234328

(43)Date of publication of application : 31.08.2001

(51)Int.Cl.

C23C 14/06
B23B 27/14
B32B 15/04

(21)Application number : 2000-049530

(71)Applicant : TOSHIBA TUNGALOY CO LTD

(22)Date of filing : 25.02.2000

(72)Inventor : KOBATA MAMORU

WATANABE TOSHIYUKI
SEKI KATSUHIKO

(54) COMBINED HARD COATING MEMBER

(57)Abstract:

PROBLEM TO BE SOLVED: To solve the problem that a conventional combined hard coating member composed of a Ti-Al-containing compound is inferior, because of many defects and strain of the coating itself, in the adhesion between the coating and a material adjacent to it and is liable to suffer peeling of the coating and is also inferior in the various properties of the coating itself and, for example, in the case of use as a cutting tool, this member rapidly causes the development of peeling, fine chipping, wear, or the like, due to the defects and strain of the coating itself and has only a short life.

SOLUTION: In the member coated with a combined hard coating, the surface of a base material is coated with a single-layer or a laminated coating layer including a combined hard coating of at least one kind among titanium- and aluminum-containing compound nitrides, compound carbides, compound carbonitrides, compound nitroxides, compound carboxides and compound carbonitroxides. Moreover, when X-ray diffraction is performed using a copper target on the surface of this coating, the following inequalities are satisfied: $h(200)/h(111) \geq 4.0$, where $h(200)$ and $h(111)$ represent the peak heights of the (200) crystal plane and the (111) crystal plane, respectively; and $1.5 \geq d(200)/d(111) \geq 0.8$, where $d(200)$ represents the half-width of the peak of the (200) crystal plane and $d(111)$ represents the half-width of the peak of the (111) crystal plane.

LEGAL STATUS

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the compound hard film covering member with which the enveloping layer containing the compound hard film which comes by the CHITAN aluminum content conjugated compound on the base material of a metal, an alloy, a sintered alloy, a ceramic sintered compact, or a super-elevated-temperature high-pressure sintered compact was covered.

[0002]

[Description of the Prior Art] The hard film is covered from the former using chemical vapor deposition (henceforth a "CVD method"), physical vapor deposition (henceforth "PVD"), or a plasma-CVD method on the base material of a metal, an alloy, a sintered alloy, a ceramic sintered compact, or a super-elevated-temperature high-pressure sintered compact, and the hard film covering member which used a base material and the hard film effectively has been used. Now, the quality of the material of the hard film in the hard film covering member currently used can mention Ti element content hard film, such as a nitride of Ti, carbon nitride, and carbide, Ti-aluminum element content compound hard film, such as a compound nitride of content of Ti and aluminum, and compound carbon nitride, and the aluminum-oxide hard film as an example of representation.

[0003] Among these hard film covering members, the Ti-aluminum element content compound hard film is covered on a base material, the property of the Ti-aluminum element content compound hard film is pulled out effectively, and many compound hard film covering members which were going to attain the longevity life are proposed. To the typical thing proposed among these about making a longevity life attain from the crystal structure of the compound hard film JP,8-209335,A, a publication-number No. 291353 official report, JP,9-295204,A, JP,9-300105,A, JP,9-300106,A, JP,9-323204,A, JP,9-323205,A, JP,10-76407,A, JP,10-76408,A, There are JP,11-1762,A, JP,11-131214,A, JP,11-131215,A, JP,11-131216,A, and JP,11-131217,A. Moreover, JP,52-28478,A is one of those typical which are proposed about the covered cemented carbide which covered not the Ti-aluminum element content compound hard film but Ti element content compound hard film.

[0004]

[Problem(s) to be Solved by the Invention] The inside of the advanced-technology reference about the compound hard film covering member which covered the Ti-aluminum element content compound hard film, JP,8-209335,A, JP,9-295204,A, JP,9-300105,A, JP,9-300106,A, JP,9-323204,A, JP,9-323205,A, To JP,10-76407,A, JP,10-76408,A, JP,11-131215,A, and JP,11-131217,A The ratio of the peak height of the crystal face to the peak height (200) of the crystal face in the X diffraction (111) of the Ti-aluminum element content compound hard film is indicated about the covering member including the requirements for a configuration which are 1 or more, 1.5 or more, and 2 or more. Moreover, the ratio of the peak height of the crystal face to the peak height (200) of the crystal face in the X diffraction (111) of the Ti-aluminum element content compound hard film is indicated by JP,9-291353,A, JP,11-131214,A, and JP,11-131216,A among advanced-technology reference about the covering member including the requirements for a configuration which are two or less.

[0005] moreover, to JP,10-317123,A The inside of the diffraction peak of the compound hard film represented by Cu and N in the X diffraction which makes K alpha rays a line source (Ti, aluminum), About a compound hard film member including the requirements for a configuration from which the crystal face serves as the highest diffraction peak height, further (200) To JP,11-1762,A It is indicated about the compound hard film covering member which makes the highest diffraction peak height the requirements for a configuration at the angle of diffraction in 42.5 - 44.5 degrees (2theta) among the diffraction peaks of this compound hard film in the X diffraction which makes Cu and K alpha rays a line source.

[0006] Having made stable cutting and a longevity life possible is indicated by the open patent official report of these 15 affairs by demonstrating control of the intergranular fracture in the compound hard film, improvement in the adhesion of a base material and the compound hard film, and wear-resistant improvement in consideration of the residual compression stress in the compound hard film in the Ti-aluminum element content compound hard film, or the crystal orientation in the compound hard film. However, the compound hard film covering member indicated by this official report of these 15 affairs From the defect of a crystal and distortion which exist in the compound hard film not being considered It cannot be satisfied with the reinforcement of the compound hard film itself, and abrasion resistance, and cannot be satisfied with adhesion with other film which, as a result, adjoins the adhesion of a base material and the compound hard film, and the compound hard film. The variation in a life is large, and when it uses as a cutting tool, it has the problem that it is difficult to be stabilized in a wide range field from a low-temperature field to an elevated-temperature field, and to obtain a longevity life.

[0007] In addition, although it is not the advanced-technology reference about the Ti-aluminum element content compound hard film, the X diffraction line in the crystal face (200) of the hard film is indicated by JP,52-28478,A by 2theta about the hard film member whose half peak width is 0.4 degrees or more. The hard film member of an indication in this official report is the hard film by PVD, and half peak width is expressing the difference with the hard film by the CVD method. From not considering the defect of the crystal of the hard film, and distortion, like the compound hard film mentioned above, the reinforcement of the hard film, It cannot be satisfied with abrasion resistance and adhesion, and the variation in a life is large, and when it uses as a cutting tool, it has the problem that it is difficult to be stabilized in a wide range field from a low-temperature field to an elevated-temperature field, and to obtain a longevity life.

[0008] This invention is what solved the above troubles. Specifically The defect, the distortion, the crystal structure, and crystal orientation of a crystal of the compound hard film which becomes by the conjugated compound containing CHITAN aluminum are considered. Especially, expand the use field as a cutting tool and the variation in the property of the compound hard film is controlled. The compound hard film with high toughness, high degree-of-hardness nature, abrasion resistance, oxidation resistance, thermal shock resistance, defect resistance, and joining-proof nature, It aims at offer of the compound hard film covering member which made the longevity life attain further by considering as the compound hard film which raised especially high intensity and peeling resistance, and raised joining-proof nature with **-ed material.

[0009]

[Means for Solving the Problem] The research on membrane formation of the hard film concerning [this invention persons] a CVD method, PVD, and plasma PVD, The result which continued at the long period of time and has done research on the hard film especially by PVD, If improvement in the plasma consistency at the time of membrane formation of the compound hard film of Ti-aluminum content and improvement in ionization efficiency are performed, gaseous-phase method epitaxial growth is carried out further and orientation of the crystal is carried out the optimal also in the hard film The defect of the crystal of the compound hard film is controlled [that the distortion in the compound hard film is eased by homogeneity,], It becomes possible, since the compound hard film of a fine crystal is obtained to raise the reinforcement, the abrasion resistance, the oxidation resistance, and thermal resistance of the compound hard film itself. The 1st knowledge that improvement in adhesion with the compound hard film, a base material or a substrate layer, or an outer layer becomes remarkable, and when the hard film

resembles a perfect crystal in this way It comes to acquire the 2nd knowledge that it is simple to judge from the height ratio and half-peak-width ratio including the highest peak in an X diffraction of an at least two diffraction line peak. Based on these knowledge, it comes to complete this invention.

[0010] The compound nitride with which the compound hard film covering member of this invention contains titanium and aluminum on the surface of a base material, It is covered as an enveloping layer of the monolayer containing at least one sort of compound hard film in compound carbide, compound carbon nitride, a compound nitric oxide, a compound carbonation object, and a compound charcoal nitric oxide, or a laminating. When an X diffraction is carried out using a copper target from the front face of this compound hard film (200) When the peak height of the crystal face is set to h (200) and the peak height of the crystal face (111) is set to h (111) It consists of $h(200) / h(111) \geq 4.0$, and when half peak width of the peak of the ** (200) crystal face is set to d (200) and half peak width of the peak of the ** (111) crystal face is set to d (111), it consists of $1.5 \geq d(200) / d(111) \geq 0.8$.

[0011] By the compound hard film covering member of this invention strengthening orientation of the crystal face in the compound hard film (200) containing titanium and aluminum using gaseous-phase method epitaxial growth, and controlling the distortion in the compound hard film as much as possible While raising the reinforcement of the compound hard film itself, and toughness, when the SHINAJI effectiveness that abrasion resistance is also excellent is demonstrated and an X diffraction is carried out using a copper target from the front face of the compound hard film When are set to $h(200) / h(111) < 4.0$ and being set to $d(200) / d(111) > 1.5$, or $d(200) / d(111) < 0.8$ (200) Since the stacking tendency to the crystal face is weak, the defect in the film and distortion become large and the above-mentioned SHINAJI effectiveness becomes weak, it is determined as the above peak height ratios and a half-peak-width ratio.

[0012]

[Embodiment of the Invention] The base material in the compound hard film covering member of this invention It is possible to use it, if it is the ingredient or matter which can bear the temperature heated when covering an enveloping layer. Specifically For example, the metal member represented by stainless steel, a heat-resistant alloy, high-speed steel, die steel, Ti alloy, and aluminum alloy, Cemented carbide, a cermet, the sintered alloy represented by the P / M high speed steel, an aluminum₂O₃ system sintered compact, an Si₃N₄ system sintered compact, a sialon system sintered compact, a ZrO₂ system sintered compact, the ceramic sintered compact represented by the silicon carbide system sintered compact, The super-elevated-temperature high-pressure sintered compact represented by a cubic boron nitride system sintered compact and the diamond system sintered compact can be mentioned. When using as a base material the ingredient or matter used as the tool for cutting, or a tool for antifriction from the former among these, it is desirable from the effectiveness of the improvement in a life as the tool for covering cutting or a tool for covering antifriction becoming high.

[0013] In using cemented carbide as a base material among these base materials The joint phase which a principal component becomes with Co and/or nickel 3 - 15 % of the weight, If it is made the cemented carbide containing at least one sort of hard phases chosen from the tungsten carbide of the remainder or tungsten carbide, 4a and 5a of the periodic table, and the carbide of 6a group metal, carbon nitride, and these mutual solid solutions It is especially desirable from becoming possible to demonstrate properties, such as reinforcement of a base material, toughness, and abrasion resistance, and the property of the compound hard film the optimal, and the improvement effectiveness in a life as a cutting tool becoming remarkable. The joint phase and hard phase which constitute the cemented carbide at this time can use what consists of a presentation component of the joint phase contained in conventional cemented carbide, and a hard phase.

[0014] Moreover, in using a ceramic sintered compact as a base material, when it uses as a base material the silicon nitride system sintered compact which becomes 80 - 98 % of the weight from the sintering acid of the remainder about silicon nitride and/or sialon, it becomes possible from mechanical properties, such as reinforcement of a base material, toughness, abrasion resistance, and thermal conductivity, and a physical characteristic, the mechanical property of the compound hard film, and a physical characteristic to demonstrate the adjustment of a base material and the compound hard film the

optimal, the improvement effectiveness in a life as a cutting tool becomes remarkable, and it is especially desirable. When the silicon nitride which constitutes the silicon nitride system sintered compact at this time becomes with alpha silicon nitride and/or beta silicon nitride, The case where sialon becomes by alpha sialon and/or beta sialon is sufficient. Sintering acid is what can use the matter contained in the silicon nitride system sintered compact or the sialon system sintered compact from the former. As concrete sintering acid For example, at least one sort of sintering acid chosen from the oxide, the magnesium oxide, the oxidization hafnium, the zirconium dioxide, the aluminum oxide, the alumimium nitride, the oxidization silicon, and these mutual solid solutions of rare earth elements, such as Y, La, Ce, and Dy, can be mentioned.

[0015] Furthermore, when using as a base material the super-elevated-temperature high-pressure sintered compact represented by a diamond system sintered compact and the cubic boron nitride system sintered compact, it is that the case where it becomes with the diamond system sintered compact which consists of a diamond more than 70 volume % and a grain boundary phase of the remainder, and the cubic boron nitride system sintered compact which consists of the cubic boron nitride and the grain boundary joint phase more than 20 volume % is desirable. The metal with which the diamond (it is hereafter described as "DIA") contains the diamond system sintered compact among these in the diamond system sintered compact of the former [phase / of 85 - 98 volume % and the remainder / grain boundary]. It is specifically desirable from being able to demonstrate both the properties of a base material and the compound hard film the optimal, and the improvement in a life as a cutting tool becoming remarkable, when at least one sort in Co, nickel, Fe, Si, and these mutual solid solutions is included, an alloy and. The grain boundary joint phase of 35 - 95 volume % and the remainder a cubic boron nitride system sintered compact 4a and 5a of the periodic table, the carbide of 6a group element, [cubic boron nitride] A nitride, boride, the nitride of Si, Mg, and aluminum, boride, oxides, and these mutual solid solutions, When consisting of at least one sort chosen from the metal of Co, nickel, Ti, and aluminum, the alloy, and the intermetallic compound, it is desirable from being able to demonstrate both the properties of a base material and the compound hard film the optimal, and the improvement in a life as a cutting tool becoming remarkable.

[0016] When there is surface precision of a base material and surface precision of a base material is made high as a problem common to these base materials, and the surface precision of the compound hard film also becomes high, for example, it is used as a cutting tool, it is desirable from frictional resistance becoming low, the dry area of a compound hard film front face and a **-ed material front face being controlled, and the improvement effectiveness in a life becoming high. The surface precision of a base material has desirable 0.1 micrometers or less at Ra which is the center line average of roughness height in the surface roughness specified to JIS B0601, and, as for more desirable one, Ra consists of 0.05 micrometers or less.

[0017] The configuration of the enveloping layer containing the compound hard film covered by these base material front faces The substrate layer which adjoins a base material and is covered for the purpose of adhesion, the interlayer who becomes by the compound hard film in this invention which adjoins this substrate layer and is covered, On the configuration which carries out a laminating more than two-layer, and a concrete target, the outermost layer covered the distinction before and behind use on the outer layer which adjoins this interlayer and is covered, and the front face of this outer layer, and for the purpose of an ornament A configuration for example, the enveloping layer by which sequential covering is carried out on the surface of a base material -- a base material -- the laminating which consists of the - substrate layer-compound hard film (interlayer)-outer layer-outermost layer The configuration of the laminating which consists of a base material-substrate layer-compound hard film (interlayer)-outer layer, the configuration which consists of a laminating of the base material-substrate layer-compound hard film (interlayer), The configuration of the laminating which consists of the base material-substrate layer-compound hard film (interlayer)-outermost layer, The configuration of the laminating which consists of the base material-compound hard film (interlayer)-outer layer-outermost layer, the configuration of the laminating which consists of a base material-compound hard film (interlayer)-outer layer, the configuration that consists of a laminating of the base material-compound

hard film (interlayer)-outermost layer, or the configuration of the base material-compound hard film (interlayer) can be mentioned. It is desirable from that there is no complicatedness of the process at the time of manufacture in a configuration of covering the compound hard film directly on a base material front face among these, becoming compaction of process time amount, and the variation on quality control decreasing.

[0018] A substrate layer becomes with a metal, an alloy, an intermetallic compound, or metallic compounds among these enveloping layers. Specifically For example, 4a and 5a of the intermetallic compound of the metals of Ti, aluminum, nickel, Co, and W, these mutual alloys, Ti-aluminum, Ti-nickel, Ti-Co, aluminum-nickel, aluminum-Co, Co-W, Ti-aluminum-nickel, and Ti-aluminum-Co, and the periodic table, the carbide of 6a group metal, a nitride, a carbonation object, The case where it becomes by at least one sort of monolayers or the multilayer chosen from a nitric oxide and the metallic compounds of these mutual solid solutions can be mentioned. An outer layer specifically Moreover, for example, 4a and 5a of the periodic table, carbide of 6a group metal, A nitride, a carbonation object, nitric oxides, these mutual solid solutions, the oxide of aluminum, a nitride, an acid nitride, a diamond, hard carbon (called diamond-like carbon), The case where it becomes by at least one sort of monolayers or the multilayer chosen from cubic boron nitride, hard boron nitride, and two or more sorts of such mixture can be mentioned. Furthermore, specifically, the outermost layer can mention the case where it becomes by at least one sort of monolayers or the multilayer chosen from the nitride of 4a, 5a, and 6a group metal, carbon nitride, nitric oxides, and these mutual solid solutions that what is necessary is just the enveloping layer in which the distinction before and behind use has easy color, and an enveloping layer with decorative effectiveness.

[0019] The compound hard film used as the main point of this invention has the case of a configuration of being covered as an interlayer like the configuration of the above-mentioned enveloping layer, when it is the configuration with which an above-mentioned substrate layer and the above-mentioned compound hard film are covered by the base material front face when becoming with the configuration with which only the compound hard film was covered by the base material front face, if another expression is carried out when the enveloping layer itself becomes by the compound hard film. If a chemical formula indicates the presentation component of this compound hard film, and membranous quality as concrete instantiation N, C (C (Ti, aluminum), N) (Ti, aluminum), (Ti, aluminum) (Ti, aluminum) (C (Ti, aluminum), N, O) (N, O) (C (Ti, aluminum), O) N, C (C (Ti, aluminum, M), N) (Ti, aluminum, M), (Ti, aluminum, M) (Ti, aluminum, M) And (Ti, aluminum, M) (C, N, O) can mention the case where it becomes in at least one sort of monolayers or the laminating chosen from inside (C (Ti, aluminum, M), O). (N, O) (However, the case of M where express one or more sorts of the element of the metal except Ti and aluminum and semimetal, and it consists of at least one sort in 4a and 5a of the periodic table, 6a group element, rare earth elements, Mn element, Mg element, Si element, and B element especially is desirable)

[0020] When the metallic element contains only Ti and aluminum, these compound hard film compound hard film [of w which can be expressed with the following chemical formula (Cx, Ny, Oz) (Tia, Alb)] [-- however The atomic ratio of Ti (titanium) element in a metallic element and b The atomic ratio of aluminum (aluminum) element in a metallic element, The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / in z / the atomic ratio of the oxygen (O) element in a nonmetallic element / the sum total of a metallic element in w] of a nonmetallic element is expressed. respectively -- a+b -- = -- one -- 0.8 -- >= -- a -- >= -- zero . -- four -- x+y+z -- = -- one -- 0.5 -- >= -- x -- >= -- zero -- one -- >= -- y -- >= -- zero . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- w -- >= -- 0.7 -- relation -- it is --] -- becoming -- a case -- *** -- It is desirable from excelling in the reinforcement of the compound hard film itself, abrasion resistance, and toughness, and there being few distortion and defects moreover and excelling in peeling resistance.

[0021] moreover, in becoming by the compound hard film containing metallic elements other than Ti and aluminum compound hard film [of w which can be expressed with the following chemical formula (Cx, Ny, Oz) (Tia, Alb, M1-a-b)] [-- however The atomic ratio of Ti (titanium) element in a metallic

element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, M 4a and 5a of the periodic table, 6a group element, rare earth elements, Si element, Mn element, At least one sort in Mg element and B element is expressed. x The atomic ratio of the carbon (C) element in a nonmetallic element, The atomic ratio of the nitrogen (N) element in a nonmetallic element and z y The atomic ratio of the oxygen (O) element in a nonmetallic element, w -- a metallic element -- the sum total -- receiving -- a nonmetallic element -- an atomic ratio -- expressing -- each -- 0.8 -- >= -- a -- >= -- zero . -- four -- 0.6 -- > -- b -- > -- zero . -- two -- x+y+z -- = -- one -- 0.5 -- >= -- x -- >= -- zero -- one -- >= -- y -- >= -- zero . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- w -- >= -- 0.7 -- relation -- it is --] -- becoming -- a case -- **** -- the reinforcement of the compound hard film itself -- It is desirable from excelling in abrasion resistance and toughness, and there being few distortion and defects moreover and excelling in peeling resistance.

[0022] When the compound hard film strengthening matter which becomes the interface of the crystal grain child of the compound hard film from a metallic element contains these compound hard film in ultralow volume, it is [that the reinforcement of the compound hard film itself and toughness are further excellent, that distortion is eased, and] desirable from improvement in peeling resistance becoming remarkable. The compound hard film strengthening matter at this time is [raising the adjustment of a base material and the compound hard film, and] desirable from raising adhesion, when consisting of a metallic element which constitutes the base material. Before covering an enveloping layer, it is also possible to form with plating, a vacuum deposition method, etc. and to diffuse this, but when this compound hard film strengthening matter diffuses the metallic element which constitutes the base material in the compound hard film, it is a desirable thing from being obtained simply.

[0023] The content of aluminum element to the sum total content of the metallic element of Ti and aluminum in the compound hard film is increasing these compound hard film from the base material front face toward the front face of the compound hard film, If another expression is carried out, the content of Ti element will increase from the front face of the compound hard film toward a base material front face, When it is made the so-called compound hard film of an inclination presentation, the adhesion of a base material and the compound hard film is excellent, It is desirable from excelling in the reinforcement of the compound hard film itself, and toughness, and the oxidation resistance of that a defect, distortion, and residual stress decrease and a compound hard film front face, abrasion resistance, and corrosion resistance being excellent. Though the increment in aluminum element at this time and Ti element has increase and decrease in micro stair-like and in the shape of [of a saw] a cutting edge, when it increases gradually in macro, the case where it is increasing continuously parabolic and in the shape of a straight line is sufficient as it.

[0024] This compound hard film is a desirable thing from the collapse-proof reinforcement from the front face of the compound hard film improving, and excelling in peeling resistance and minute-proof chipping nature, when the columnar crystal which grew perpendicularly in the shape of a column to the base material front face, considering the structure of the compound hard film itself is contained.

Specifically, the compound hard film containing this columnar crystal can illustrate the case where minute amount content of the compound hard film strengthening matter mentioned above in each of these granular crystals and columnar crystals is carried out, when the whole compound hard film becomes in the layer of a columnar crystal, it becomes in the intermingled layer of a granular crystal and a columnar crystal and it becomes in the laminating of the layer of a granular crystal, and the layer of a columnar crystal. When the compound hard film strengthening matter contains below 1 volume % preferably below 3 volume % to the sum total of the compound hard film and the compound hard film strengthening matter, while excelling in the collapse-proof reinforcement of the from both [a perpendicular direction and / horizontal] a front face, and compressive strength-proof among these, it is desirable from the ability to demonstrate the SHINAJI effectiveness of excelling also in abrasion resistance. [of the compound hard film] [both]

[0025] As for these compound hard film, it is desirable to consider the structure of the compound hard film itself by in which location of the configuration of an enveloping layer mentioned above it exists. As a configuration of this enveloping layer After covering a substrate layer to a base material when

covering the direct compound hard film to a base material or, The compound hard film member by the 1st configuration for which the compound hard film is covered in a substrate layer, and the front face of the compound hard film is used in the condition of contacting other matter (for example, **-ed material in a cutting tool), When an outer layer is covered on the front face of the compound hard film, an outer layer and the outermost layer can be covered on the front face of the compound hard film, and it can divide roughly into the compound hard film member by the 2nd configuration for which the front face of the compound hard film is used in the condition of not contacting other matter and directly.

[0026] In the case of the compound hard film member by the 1st configuration, among these By center line average-of-roughness-height Ra in the surface roughness specified to JIS B0601, 0.1 micrometers or less, if the front face of the compound hard film makes it 0.05 micrometers or less preferably When it is used as a cutting tool, from that the damage to **-ed material is eased, that cutting force is eased, and discharge of a chip becoming easy, it becomes long lasting and is much more desirable thing. Moreover, also in the compound hard film member by the 2nd configuration, although it changes with the film thickness of an outer layer, or sum total film thickness of an outer layer and the outermost layer, when surface roughness of the compound hard film is made above, the surface roughness of an outer layer and the outermost layer is also a desirable thing from it being smooth, becoming flat and being able to demonstrate the same effectiveness as ****.

[0027] Although the film thickness of each class which constitutes an enveloping layer is chosen by the configuration of an application, a configuration, and an enveloping layer In the case of the compound hard film member by the 1st above-mentioned configuration The subject of an enveloping layer becomes the compound hard film, and in this case, 1-20 micrometers, in making a substrate layer intervene mainly for the purpose of adhesion, the film thickness of the compound hard film When film thickness of a substrate layer is set to 0.2-2 micrometers, it is desirable from the reinforcement, the abrasion resistance, the toughness, and peeling resistance of the enveloping layer itself. Moreover, in becoming by the compound hard film member by the 2nd above-mentioned configuration, when it sets [the film thickness of a substrate layer / the film thickness of 0.2-2 micrometers and the compound hard film] film thickness of 1-10 micrometers and the outermost layer to 0.5-2 micrometers for 1-10 micrometers and the film thickness of an outer layer, it is desirable from the ability to demonstrate the property of each membrane layer the optimal. At first, when becoming with stoichiometric composition, the case where it becomes with non-stoichiometric composition is sufficient as a substrate layer, an outer layer, and the outermost layer, and they consist substantially of non-stoichiometric composition the compound hard film explained in full detail above in many cases.

[0028] The compound hard film member of this invention which becomes with the above gestalten It is a thing usable for various kinds of applications. Specifically for example, as the intermittence cutting tool which a lathe-turning tool, a milling cutter tool, a drill, the cutting tool represented by the end mill, especially **-ed material are a casting and steel, and needs shock resistance, or a rotation cutting tool From mold tools, such as a dice and punch, as tools for antifriction, such as cutting cutting edges, such as a slitting machine, and a decision cutting edge It is usable as an engineering-works construction tool represented by the cutting tool used for a mine, a road, civil engineering works, etc., a digging tool, a **** tool, and the crushing tool as tools for corrosion-proof antifriction, such as a nozzle and a tool with **. Among these, the compound hard film member of this invention is a desirable thing from demonstrating the property of the compound hard film the optimal, when using it as cutting tools, such as rotation cutting tools, such as a cutting tool with which temperature, friction, a thermal shock, a compression impact, etc. serve as a severe condition most in micro especially a drill, and an end mill, and a throwaway tip. When using this compound hard film member as a cutting tool, when the film thickness of the compound hard film is formed so that it may decrease toward the ridgeline section formed in the cutting edge of a cutting tool, it is a desirable thing from excelling in peeling resistance and minute chipping nature. Moreover, being formed so that the film thickness of the enveloping layer containing these compound hard film may decrease toward the ridgeline section formed in the cutting edge of a cutting tool also makes the same effectiveness caused, and it is a desirable thing.

[0029] The metal member represented by the stainless steel with which the compound hard film

covering member of this invention is marketed from the former, a heat-resistant alloy, high-speed steel, die steel, Ti alloy, and aluminum alloy, Cemented carbide, a cermet, the sintered alloy represented by the P / M high speed steel, an aluminum₂O₃ system sintered compact, an Si₃N₄ system sintered compact, a sialon system sintered compact, a ZrO₂ system sintered compact, the ceramic sintered compact represented by the silicon carbide system sintered compact, The super-elevated-temperature high-pressure sintered compact represented by a cubic boron nitride system sintered compact and the diamond system sintered compact is used as a base material. Although an enveloping layer can be covered with the PVD, CVD method, or plasma-CVD method currently performed from the former on a base material and it can produce after grinding the front face of this base material if needed and performing ultrasonic cleaning, organic solvent washing, etc. When it produces by the following approaches, the property and adhesion of that the gaseous-phase epitaxial crystal growth and crystal orientation of that improvement in a plasma consistency and improvement in ionization efficiency are attained and the compound hard film itself become easy and the compound hard film are a desirable thing from excelling more.

[0030] When the important description is concretely explained in full detail as the manufacture approach for obtaining this compound hard film covering member, the front face of a base material At least one sort of mechanical processes in the blasting processing currently performed from the former, shot-peening processing, polish processing, and barrel processing, At least one sort of chemical preparation in washing by the surface corrosion by the electrolytic etching by the acid or alkaline electrolytic solution, the acid solution, and the alkali solution or water, and the organic solution, When processing chosen from coincidence or the art performed separately in this mechanical process and chemical preparation is performed, it is desirable from the ability to control [that the defect on the front face of a base material is removable, that the adhesion of the compound hard film can be raised, that the distortion in the film can be controlled, and] the defect in the film. Moreover, a base material is that it is desirable to add such mechanical treatment and/or chemical preparation, and heat treatment by low temperature, and to also heighten above-mentioned effectiveness.

[0031] When covering the compound hard film on the surface of a base material, it is desirable to carry out by the PVD represented by a sputtering technique and the ion plating method, and it is especially desirable among these from adjustment of the compound hard film being easy when it carries out by the magnetron sputtering method or the arc-plasma ion plating method. When arranging a base material in the reaction container of an ion plating system, specifically carrying out bombardment processing of the base material front face and bombardment processing by metallic element ion or bombardment processing by both metallic element ion and nonmetallic element ion is performed, it is desirable from above-mentioned effectiveness being heightened. When you need the substrate layer of a metal, an alloy, or an intermetallic compound among the above-mentioned substrate layers and it especially gives ion bombardment containing metallic element ion, it is desirable from that formation of a substrate layer is easy, and the adhesion of a base material and a substrate layer becoming high.

[0032] The covering conditions of the compound hard film need to attach importance to the effect of equipment itself, such as structure of a reaction container, and adjustment of the plasma. Specifically for example, the high voltage -- (-- using the equipment which carries out plasma generating of the ion for the pulse-like high voltage and a RF with acceleration with the power source of addition) depending on the case, and the equipment which can adjust the plasma by the field -- In addition, the thing for which it is necessary to consider about arrangement of the ambient pressure force in a reaction container, temperature, an arc discharge current . electrical potential difference, base material bias voltage, and a sample etc., and especially an arc discharge electrical potential difference is made high to the conventional conditions among these, Rotations, vertical movement, etc. of making base material bias voltage high and a sample are important requirements.

[0033]

[Operation trial 1] An operation trial explains the operation gestalt of this invention explained in full detail above as a still more concrete example of representation. First, the trial which covered the direct compound hard film on these base material front faces is explained using the base material of cemented

carbide and the base material of a cermet which were produced through each process of the conventional combination, mixing, shaping, and sintering. The base material 1 of the cemented carbide of SNGN120408 configuration - the base material 5, and the base material 6 of a cermet by the ISO standard produced by Table 1 shown of the combination presentation component are used for a base material. A grinding process is performed for the vertical side and peripheral face of these base materials by the diamond wheel of 270#. After performing -25 degreex0.10mm honing to the edge-of-a-blade section by the 400# diamond wheel and performing wet blasting processing, washing processing, and desiccation processing for a front face further, the compound hard film was covered with the arc ion plating system.

[0034] Processing conditions covered the compound hard film, after carrying out bombardment processing of each base material front face in a reaction container. Ambient atmosphere: vacuum [in a reaction container], base material temperature: 873K, and arc current: 70A, base material bias voltage: -600V, and Ar gas bombardment performed bombardment processing, covering of the compound hard film -- quantity-of-gas-flow [in a reaction container]: -- 200 - 350SCCM, an evaporation source: Ti-aluminum alloy, arc voltage: 200-300V, arc current: 150-200A, base material temperature: 773-873K, and base material bias voltage: -100--200V performed, and this invention article 1-6 which covered the compound hard film on each front face of the base materials 1-6 shown in Table 1, and was shown in Table 2 was obtained. this invention article 1 the ambient atmosphere in a reaction container among these An Ar-N₂-O₂ gas presentation, An Ar-N₂-CO gas presentation and the other this invention articles 3-6 carry [this invention article 2] out by Ar-N₂ gas presentation. To the evaporation source of this invention article 4 It carried out by having changed from the alloy with many Ti elements to the alloy with many aluminum elements, and carried out by changing to an alloy with many alloy-Ti elements with many alloy-aluminum elements with many Ti elements to the evaporation source of this invention article 5.

[0035] The comparison article 1-4 which covered the compound hard film as a comparison on each front face of the base materials 3-6 shown in Table 1, and was shown in Table 2 was obtained. The base material front face of use in the above-mentioned this invention article 1-6 was [the base material front face of use in the comparison article 1-4 of base material surface roughness] Ra=0.1-0.05micrometer to Ra=0.01-0.005micrometer. Except for wet blasting processing, others processed almost similarly the base material processing in the comparison article 1-4 among the base material surface preparation of this invention article mentioned above. Moreover, arc-voltage: 10-50V among the processing conditions of the compound hard film of this invention article which mentioned above covering of the compound hard film in this comparison article 1-4, an arc current: It processed almost similarly except having been referred to as 200-250A, and base material bias voltage: -30--80V. However, what has a fixed Ti-aluminum element ratio was used for the evaporation source at the time of the compound hard membrane process of the comparison article 1-4.

[0036] in this way, about each compound hard film of the obtained this invention article 1-6 and the comparison article 1-4 X-ray diffractometer, a scanning electron microscope, a metaloscope, EDS equipment, a Vickers hardness tester, and the scratch testing machine that scratches and is equivalent to a hardness tester are used. It asked for the content of Ti and aluminum of h (200) by the X diffraction from a compound hard film front face / d [h (111) and] (200)/d (111), and a compound hard film front face, the hardness of a compound hard film front face, and scratch reinforcement, and each result was shown in Table 2. In addition, the compound hard film thickness of this invention article 1-6 and the comparison article 1-4 consisted of about 5-7 micrometers, the granularity of the compound hard film front face of this invention article 1-6 was Ra=0.015-0.010micrometer mostly, and the granularity of the compound hard film front face of the comparison article 1-4 was Ra=0.15-0.10micrometer mostly. Moreover, the oxygen element in a nonmetallic element of the compound hard film of this invention article 1 was ***** not more than 1at% (Ti, aluminum) (N, O), the oxygen element in a nonmetallic element of the compound hard film of this invention article 2 was a charcoal nitride not more than 2at% (Ti, aluminum) (N, C), and other compound hard film was nitrides displayed by N (Ti, aluminum).

[0037] Subsequently, the following cutting conditions performed the wet intermittence cutting trial

using this invention article 1-5 and the comparison article 1-3. cutting conditions -- S45C of **-ed material: machine structural-carbon-steel material – four slot ON round bar, cutting speed: 150 m/min, and delivery: -- 0.3 mm/rev, it cut deeply and :2.0mm, tool configuration:SNGN120408, and water soluble cutting oil performed. When the chipping of a cutting edge and an enveloping layer exfoliated, the result of a wet intermittence cutting trial made the tool life the time of the amount of average flank wear or the amount of groove wear amounting to 0.3mm, found each time amount at that time which can be cut, and wrote it together to Table 2 as a life ratio to the life of the comparison article 1.

[0038]

[Table 1]

試料番号	焼結合金の組成成分 (配合時)	重量%
基材 1	97WC-3Co	
基材 2	91WC-3TaC-1TiC-5Co	
基材 3	88WC-2TaC-2TiC-8Co	
基材 4	86.5WC-1TaC-0.5NbC-2TiC-10Co	
基材 5	80WC-1Cr3C2-1VC-18Co	
基材 6	26TiC-26TiN-20WC-10TaC-1Mo2C-1ZrC-8Ni-8Co	

[0039]

[Table 2]

試料 番号	複合硬質膜の 結晶面ピーク		複合硬質膜 の成分比、特性			切削試験 寿命比	
	高さ比 : h (200)/(111)	半価幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スクラッチ 強度(N)		
本 発 明 品	1	5.0	0.85	52 : 48	2780	70	2.5
	2	4.8	0.9	55 : 45	2950	75	2.8
	3	7.5	1.1	53 : 47	2900	90	5.2
	4	5.3	1.4	58 : 44	2920	86	4.8
	5	5.8	1.2	58 : 42	3000	80	3.5
	6	6.5	1.3	50 : 50	2880	88	なし
比 較 品	1	3.7	1.2	53 : 47	2910	43	1.0
	2	1.4	2.3	50 : 50	2950	34	0.9
	3	2.4	2.5	55 : 45	2850	40	1.1
	4	0.1	0.6	40 : 60	3100	25	なし

[0040]

[Operation trial 2] The cemented carbide of the base material 3 shown in Table 1 of the operation trial 1 was used as the base material, and this invention article 7-13 and the comparison article 5 which covered the enveloping layer of the laminating which contains more than two-layer [in a substrate layer, the compound hard film, an outer layer, and the outermost layer] on the front face of this base material, and the comparison article 6 were obtained using the arc ion plating system. Among these, Ti substrate layer of this invention article 7 carried out Ti vacuum evaporation to the base material front face, and the intermetallic-compound (Ti-aluminum) substrate layer of this invention article 8 was vapor-deposited to the bombardment processing and coincidence by the evaporation source (Ti-aluminum), and it vapor-deposited Ti substrate layer of this invention article 9 to the bombardment processing and coincidence by Ti evaporation source. Base material processing of this invention article 7-13 and membrane formation of the compound hard film were processed almost like this invention article 3-6 in the operation trial 1, and processed base material processing of the comparison article 5 and the comparison article 6 and membrane formation of the compound hard film almost like the comparison article 1 in the operation trial 1. The other substrate layers, outer layers, and outermost layers were processed by the almost conventional process.

[0041] in this way, about the substrate layer, the compound hard film, outer layer, and the outermost

layer of each of the obtained this invention article 7-13, the comparison article 5, and the comparison article 6 It investigated like the operation trial 1, the configuration, each membranous quality, and film thickness of each enveloping layer were shown in Table 3, and the crystal-face peak height ratio by the X diffraction in a compound hard film front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and scratch reinforcement were shown in Table 4. Moreover, about this invention article 7-13, the comparison article 5, and the comparison article 6, the cutting trial was performed like the cutting conditions of the operation trial 1, and it asked for each life ratio to the comparison article 5, and wrote together to Table 4. In addition, after surface investigation of the compound hard film covers the compound hard film, it is taken out from a reaction container and performed, and this invention article 10 heat-treated, diffused the joint phase in a base material in the enveloping layer, and made a substrate layer and the compound hard film contain the joint phase of about 1 volume % after [all] covering termination. Moreover, the charcoal titanium nitride layer of a substrate layer consists of a columnar crystal, and this invention article 11 is considered as a columnar crystal crystal in consideration of a plasma consistency, radical reaction, and a membrane formation rate at the time of membrane formation of the compound hard film.

[0042]

[Table 3]

試料番号	被覆層の膜質および膜厚さ(μm)			
	下地層	複合硬質膜	外層	最外層
本発明品	7 0.5Ti	6(Ti,Al)N	なし	なし
	8 0.5(Ti-Al)	6(Ti,Al)N	なし	1TiN
	9 0.5Ti-2TiN	6(Ti,Al)N	なし	なし
	10 1TiN	3(Ti,Al)N	5Al ₂ O ₃	なし
	11 1TiN-3TiCN	3(Ti,Al)N	5Al ₂ O ₃	1TiN
	12 1.5TiN	3(Ti,Al)N	2TiNO-3Al ₂ O ₃	1TiCN
	13 1.5TiN	4(Ti,Al)N	2TiCO-3Al ₂ O ₃	1TiN
比較品5	1TiN	3(Ti,Al)N	5Al ₂ O ₃	なし
比較品6	1.5TiN	4(Ti,Al)N	2TiNO-3Al ₂ O ₃	1TiN

[0043]

[Table 4]

試料番号	複合硬質膜の結晶面ピーカ		複合硬質膜の成分比、特性			切削試験 寿命比
	高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクレーパー 強度(N)	
本発明品	7 7.3	1.2	58:42	2900	65	5.7
	8 7.4	1.3	53:47	2950	62	4.8
	9 7.6	1.2	55:45	2900	78	5.5
	10 7.8	1.1	54:46	2910	95	7.1
	11 8.5	1.0	50:50	3000	88	6.5
	12 7.4	0.9	51:49	2890	73	5.1
	13 7.2	0.95	57:43	2920	70	4.7
比較品5	8.3	3.0	54:46	2900	35	1.0
比較品6	3.0	2.4	53:47	2930	42	1.3

[0044]

[Operation trial 3] The base material 7 of the ceramic sintered compact produced by 70 volume % aluminum₂O₃-30 volume %TiCN (combination presentation), The base material 8 of the ceramic sintered compact produced by the 70 volume %aluminum₂O₃-30 volume %SiC whisker (combination presentation), The base material 9 of the ceramic sintered compact produced by 96 volume %Si₃N₄-1

volume %MgO-1 volume %Y₂O₃-2 volume %HfO₂ (combination presentation), The base material 10 of the ceramic sintered compact produced by 90 volume %Si₃N₄-4 volume %AlN-4 volume %aluminum₂O₃-1 volume %MgO-1 volume %Y₂O₃ (combination presentation) is used. After processing the base material almost like this invention article 3-6 in the operation trial 1 and vapor-depositing Ti of about 1-micrometer film thickness on each base material front face, the compound hard film was covered and this invention article 14-17 was obtained. Moreover, using a base material 7 and a base material 9 as a comparison, it processed almost like the comparison article 1 in the operation trial 1, and the comparison article 7 and the comparison article 8 were obtained.

[0045] In this way, about the compound hard film of the obtained this invention article 14-17, the comparison article 7, and the comparison article 8, it investigated like the operation trial 1, and the crystal-face peak height ratio by the X diffraction in a compound hard film front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and scratch reinforcement were shown in Table 5. moreover, this invention article 14, this invention article 15, and the comparison article 7 -- **-ed material -- :FCD600, cutting speed:150 m/min, infeed:1.5mm, delivery:0.2mm /, cutting edge, tool configuration:SNGN120408, honing:0.15x-25 degree, and a dry type milling cutter cutting trial were performed. this invention article 16, this invention article 17, and the comparison article 8 -- **-ed material -- :FCD250, cutting speed:600 m/min, infeed:3.0mm, delivery:0.1mm /, cutting edge, tool configuration:SNGN120412, honing:0.15x-25 degree, and a dry type milling cutter cutting trial were performed. Like the cutting trial in the operation trial 1, these dry type milling cutter results made this invention article 14 and this invention article 15 the life ratio to the comparison article 7, and wrote together this invention article 16 and this invention article 17 to Table 5 as a life ratio to the comparison article 8. In addition, the compound hard film thickness of this invention article 14-17, the comparison article 7, and the comparison article 8 was about 5 micrometers.

[0046]

[Table 5]

試料 番号	複合硬質膜の 結晶面ピーク		複合硬質膜 の成分比、特性			切削試験 寿命比
	高さ比 : h (200)/(111)	半価幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本 品	1 4	6.8	60 : 40	2880	75	6.5
発 明	1 5	6.3	62 : 38	2850	68	6.1
品	1 6	12.5	48 : 52	3150	70	5.3
	1 7	13.4	45 : 55	3200	73	6.0
比較品 7	3.5	2.3	58 : 42	2950	31	1.0
比較品 8	3.7	2.1	46 : 54	3130	32	1.0

[0047]

[Operation trial 4] The base material 11 of the cubic boron nitride system sintered compact of super-elevated-temperature high pressure produced by 40 volume %cBN-5 volume %aluminum₂O₃-5 volume %AlN-10 volume %aluminum-10 volume %Mg-10 volume %B-20 volume %TiN (combination presentation), The base material 12 of the cubic boron nitride system sintered compact of super-elevated-temperature high pressure produced by 85 volume %cBN-2 volume %Co-5 volume %aluminum-2 volume %Mg-6 volume %TiN (combination presentation), The base material 13 of the DIA system sintered compact of super-elevated-temperature high pressure produced by the 95 volume %DIA-2 volume %Co-2 volume %nickel-1 volume ZrC (combination presentation), The base material 14 of the DIA system sintered compact of super-elevated-temperature high pressure produced with 97 volume %DIA-1 volume %Co-1 volume %nickel-1 volume Mg (combination presentation) is used. After having processed the base material almost like this invention article 3-6 in the operation trial 1, vapor-depositing Ti of about 1-micrometer film thickness on the front face of a base material 11 and a base material 12 and performing nickel electroless deposition of about 1-micrometer thickness to the front face of a base material 13 and a base material 14, the compound hard film was covered and this

invention article 18-21 was obtained. Moreover, using a base material 11 and a base material 13 as a comparison, it processed almost like the comparison article 1 in the operation trial 1, and the comparison article 9 and the comparison article 10 were obtained.

[0048] In this way, about the compound hard film of the obtained this invention article 18-21, the comparison article 9, and the comparison article 10, it investigated like the operation trial 1, and the crystal-face peak height ratio by the X diffraction in a compound hard film front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and scratch reinforcement were shown in Table 6. Moreover, this invention article 18, this invention article 19, and the comparison article 9 performed **-ed material: SCM415 (hardness: abbreviation HRC61), cutting speed: 150 m/min, infeed: 0.5mm, delivery: 0.1 mm/rev, tool configuration: T **** 160408, honing: 0.15x-25 degree, and a periphery continuation dry type lathe-turning trial, and wrote together the result to Table 6. Evaluation of this periphery continuation dry type lathe-turning trial was performed like the cutting trial in the operation trial 1, and it expressed as a life ratio to the comparison article 9. In addition, the compound hard film thickness of this invention article 18-21, the comparison article 9, and the comparison article 10 was about 5 micrometers.

[0049]

[Table 6]

試料 番号	複合硬質膜の 結晶面ピーカ		複合硬質膜 の成分比、特性			切削試験 寿命比
	高さ比 : h (200)/(111)	半価幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スクランチ 強度(N)	
本品 18	19.5	1.1	58 : 42	2960	85	8.2
発明品 19	18.4	1.0	59 : 41	2930	83	8.8
明品 20	8.5	1.05	55 : 45	2950	76	なし
品 21	8.9	0.95	54 : 46	2970	74	なし
比較品 9	3.1	2.0	55 : 45	2910	23	1.0
比較品 10	3.5	1.7	52 : 48	2940	25	なし

[0050]

[Effect of the Invention] The compound hard film by the crystal growth according [the compound hard film covering member of this invention] to gaseous-phase method epitaxial and crystal orientation is covered, It is [that distortion by the compound hard film itself and a defect are controlled,] a fine crystal, It contrasts with the compound hard film covering member which separated from a conventional compound hard film covering member or conventional this invention from it being the compound hard film with which compound hard film strengthening matter, such as a columnar crystal crystal and/or a metal of a minute amount, was contained depending on the case. Adhesion and peeling resistance are very excellent to the matter contiguous to a base material, the compound hard film, a substrate layer and the compound hard film, the compound hard film, and compound hard film, such as an outer layer, The high toughness, the high intensity, the thermal resistance, the thermal shock resistance, the oxidation resistance, and abrasion resistance of the compound hard film itself are excellent, As the result, for example, the thing for which the high toughness to which importance is attached as a cutting tool, abrasion resistance, thermal shock resistance, defect resistance, oxidation resistance, and joining-proof nature improve notably, and reinforcement is attained when it is used as a cutting tool, There is remarkable effectiveness that efficient-ization in cutting being attained and variation are small stable.

[Translation done.]

* NOTICES *

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. **** shows the word which can not be translated.

3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The compound nitride, compound carbide which contain titanium and aluminum on the surface of a base material, When it is covered as an enveloping layer of the monolayer containing at least one sort of compound hard film in compound carbon nitride, a compound nitric oxide, a compound carbonation object, and a compound charcoal nitric oxide, or a laminating and an X diffraction is carried out using a copper target from the front face of this compound hard film (200) When the peak height of the crystal face is set to h (200) and the peak height of the crystal face (111) is set to h (111) The compound hard film covering member which consists of $h(200)/h(111) \geq 4.0$, and consists of $1.5 \geq d(200)/d(111) \geq 0.8$ when half peak width of the peak of the ** (200) crystal face is set to d (200) and half peak width of the peak of the ** (111) crystal face is set to d (111).

[Claim 2] The above-mentioned base material is a compound hard film covering member according to claim 1 which consists of cemented carbide containing at least one sort as which 4 - 15 % of the weight and the remainder were chosen from tungsten carbide or tungsten carbide, 4a and 5a of the periodic table, and the carbide of 6a group metal, carbon nitride, and these mutual solid solutions in the joint phase which a principal component becomes with Co and/or nickel of hard phases.

[Claim 3] The above-mentioned base material is a compound hard film covering member according to claim 1 to which the remainder becomes 80 - 98 % of the weight from the silicon nitride system sintered compact which is sintering acid about silicon nitride and/or sialon.

[Claim 4] The above-mentioned base material is a compound hard film covering member according to claim 1 which consists of a super-elevated-temperature high-pressure sintered compact containing at least one sort of grain boundary joint phases as which 20 - 90 % of the weight and the remainder were chosen in cubic boron nitride from the nitride of Ti, aluminum, Mg, and Si, borides, and these mutual solid solutions.

[Claim 5] The above-mentioned base material is a compound hard film covering member given in any 1 term of claims 1-4 which the surface roughness of this base material becomes from 0.1 micrometers or less by the average surface roughness by Ra of JIS.

[Claim 6] The above-mentioned enveloping layer is a compound hard film covering member given in any 1 term of claims 1-5 which becomes by the above-mentioned compound hard film.

[Claim 7] The above-mentioned compound hard film is a compound hard film covering member given in any 1 term of claims 1-6 which consists of the cubic mold crystal structure which film thickness becomes by 1-15 micrometers.

[Claim 8] the above-mentioned compound hard film -- w(Cx (Tia, Alb), Ny, Oz) [-- however The atomic ratio of Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / in z / the atomic ratio of the oxygen (O) element in a nonmetallic element / the sum total of a metallic element in w] of a nonmetallic element is expressed. respectively -- a+b -- = -- one -- 0.8 -- >= -- a -- >= -- zero . -- four -- x+y+z -- = -- one -- 0.5 -- >= -- x -- >= -- zero --

one -- >= -- y -- >= -- zero . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- w -- >= -- 0.7 -- relation - - it is --] -- expressing -- having -- compound -- hard -- the film -- containing -- a claim -- one - seven -- some -- one -- a term -- a publication -- compound -- hard -- the film -- covering -- a member .

[Claim 9] the above-mentioned compound hard film -- w(C_x (Ti, Al, M_{1-a-b}, Ny, Oz) [- however The atomic ratio of Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, M expresses at least one sort in 4a and 5a of the periodic table, 6a group element, and Si, Mn, Mg and B. The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / in z / the atomic ratio of the oxygen (O) element in a nonmetallic element / the sum total of a metallic element in w] of a nonmetallic element is expressed. each -- 0.8 -- >= -- a -- >= -- zero . -- four -- 0.6 -- > -- b -- > -- zero . -- two -- x+y+z -- = -- one -- 0.5 -- >= -- x -- >= -- zero -- one -- >= -- y -- >= -- zero . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- w -- >= -- 0.7 -- relation -- it is --] -- expressing -- having -- compound -- hard -- the film -- containing -- a claim -- one - seven -- some -- one -- a term -- a publication -- compound -- hard -- the film -- covering - - a member .

[Claim 10] The above-mentioned compound hard film is a compound hard film covering member given in any 1 term of claims 1-9 which at least one sort of compound hard film strengthening matter chosen into this compound hard film from the metals of nickel, Co, W, Mo, aluminum, and Ti, these mutual alloys, and these intermetallic compounds contains.

[Claim 11] The above-mentioned compound hard film is a compound hard film covering member given in any 1 term of claims 1-10 in which the columnar crystal which comes to be perpendicularly pillar-shaped is contained to the front face of the above-mentioned base material.

[Claim 12] The above-mentioned compound hard film is a compound hard film covering member given in any 1 term of claims 1-11 which the content of aluminum element to the sum total content of the metallic element of Ti and aluminum in this compound hard film is increasing from the front face of the above-mentioned base material toward the front face of this compound hard film.

[Claim 13] The above-mentioned compound hard film is a compound hard film covering member given in any 1 term of claims 1-12 which the surface roughness of this compound hard film becomes from 0.1 micrometers or less by the average surface roughness by Ra of JIS.

[Claim 14] A compound hard film covering member given in any 1 term of claims 1-13 to which the thin layer which becomes with the intermetallic compound containing the metal of Ti and/or aluminum, the alloy of Ti and aluminum, Ti, and/or aluminum intervenes by the film thickness of 1 micrometer or less between the above-mentioned compound hard film and the above-mentioned base material.

[Claim 15] A compound hard film covering member given in any 1 term of above-mentioned claims 1-14 is a compound hard film covering member used as a cutting tool.

[Claim 16] The above-mentioned cutting tool is a compound hard film covering member according to claim 15 to which the film thickness of the above-mentioned compound hard film is decreasing toward the ridgeline section.

[Translation done.]

(19)日本国特許庁 (JP)

(12) 公開特許公報 (A)

(11)特許出願公開番号

特開2001-234328

(P2001-234328A)

(43)公開日 平成13年8月31日(2001.8.31)

(51)Int.Cl.⁷

C 23 C 14/06

識別記号

F I

テマコード(参考)

C 23 C 14/06

K 3 C 0 4 6

4 F 1 0 0

H 4 K 0 2 9

N

B 23 B 27/14

B 23 B 27/14

A

審査請求 有 請求項の数16 OL (全 12 頁) 最終頁に続く

(21)出願番号

特願2000-49530(P2000-49530)

(22)出願日

平成12年2月25日(2000.2.25)

(71)出願人 000221144

東芝タンガロイ株式会社

神奈川県川崎市幸区堀川町580番地 ソリ

ッドスクエア

(72)発明者 木幡 譲

神奈川県川崎市幸区堀川町580番地 ソリ

ッドスクエア 東芝タンガロイ株式会社内

(72)発明者 渡邊 敏行

神奈川県川崎市幸区堀川町580番地 ソリ

ッドスクエア 東芝タンガロイ株式会社内

(72)発明者 関 克彦

神奈川県川崎市幸区堀川町580番地 ソリ

ッドスクエア 東芝タンガロイ株式会社内

最終頁に続く

(54)【発明の名称】複合硬質膜被覆部材

(57)【要約】

【課題】従来のTi-A1を含む化合物でなる複合硬質膜の被覆部材は、複合硬質膜自体に多くの欠陥や歪みを有していることから、複合硬質膜と隣接する物質との密着性に劣ること、剥離しやすいうこと、複合硬質膜自体の諸特性が劣ること、例えば切削工具として用いる場合には、複合硬質膜自体の欠陥や歪みから剥離、微小チッピング、摩耗などが急速に発達しやすくなつて、短寿命になるという課題がある。

【解決手段】基材の表面にチタンとアルミニウムとを含む複合塗化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さをh(200)とし、(111)結晶面のピーク高さをh(111)としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅をd(200)とし、該(111)結晶面のピークの半価幅をd(111)としたときに、 $1.5 \geq d$

(200)/d(111) ≥ 0.8 からなる複合硬質膜被覆部材。

【特許請求の範囲】

【請求項1】基材の表面にチタンとアルミニウムとを含む複合窒化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さをh(200)とし、(111)結晶面のピーク高さをh(111)としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅をd(200)とし、該(111)結晶面のピークの半価幅をd(111)としたときに、 $1.5 \geq d(200)/d(111) \geq 0.8$ からなる複合硬質膜被覆部材。

【請求項2】上記基材は、主成分がC_oおよび/またはN_iでなる結合相を4~15重量%と、残部が炭化タングステンまたは炭化タングステンと周期律表の4a, 5a, 6a族金属の炭化物、炭窒化物およびこれらの相互固溶体から選ばれた少なくとも1種との硬質相を含有する超硬合金からなる請求項1に記載の複合硬質膜被覆部材。

【請求項3】上記基材は、窒化珪素および/またはサイアロンを80~98重量%と、残部が焼結助剤である窒化珪素系焼結体からなる請求項1に記載の複合硬質膜被覆部材。

【請求項4】上記基材は、立方晶窒化硼素を20~90重量%と、残部がTi, Al, Mg, Siの窒化物、硼化物およびこれらの相互固溶体の中から選ばれた少なくとも1種の粒界結合相とを含有する超高温高压焼結体からなる請求項1に記載の複合硬質膜被覆部材。

【請求項5】上記基材は、該基材の表面粗さがJIS規格のRaによる平均表面粗さで0.1μm以下からなる請求項1~4のいずれか1項に記載の複合硬質膜被覆部材。

【請求項6】上記被覆層は、上記複合硬質膜でなる請求項1~5のいずれか1項に記載の複合硬質膜被覆部材。

【請求項7】上記複合硬質膜は、膜厚さが1~15μmでなる立方晶型結晶構造からなる請求項1~6のいずれか1項に記載の複合硬質膜被覆部材。

【請求項8】上記複合硬質膜は、(Ti_a, Al_b)_{(C_x, N_y, O_z)_w} [ただし、aは金属元素中のTi(チタン)元素の原子比、bは金属元素中のAl(アルミニウム)元素の原子比、xは非金属元素中の炭素(C)元素の原子比、yは非金属元素中の窒素(N)元素の原子比、zは非金属元素中の酸素(O)元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それが $a+b=1$ 、 $0.8 \geq a \geq 0.4$ 、 $x+y+z=1$ 、 $0.5 \geq x \geq 0.1$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0.1$ 、 $0.05 \geq w \geq 0.7$ の関係にある] で表される複合硬質膜を含有する請求項1~7のいずれか1項に記載

の複合硬質膜被覆部材。

【請求項9】上記複合硬質膜は、(Ti_a, Al_b, M_{1-a-b})_{(C_x, N_y, O_z)_w} [ただし、aは金属元素中のTi(チタン)元素の原子比、bは金属元素中のAl(アルミニウム)元素の原子比、Mは周期律表の4a, 5a, 6a族元素、Si, Mn, Mg, Bの中の少なくとも1種を表し、xは非金属元素中の炭素(C)元素の原子比、yは非金属元素中の窒素(N)元素の原子比、zは非金属元素中の酸素(O)元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それが $0.8 \geq a \geq 0.4$ 、 $0.6 > b > 0.2$ 、 $x+y+z=1$ 、 $0.5 \geq x \geq 0.1$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0.1$ 、 $0.05 \geq w \geq 0.7$ の関係にある] で表される複合硬質膜を含有する請求項1~7のいずれか1項に記載の複合硬質膜被覆部材。

【請求項10】上記複合硬質膜は、該複合硬質膜中にNi, Co, W, Mo, Al, Tiの金属、これらの相互合金、これらの金属間化合物の中から選ばれた少なくとも1種の複合硬質膜強化物質が含有されている請求項1~9のいずれか1項に記載の複合硬質膜被覆部材。

【請求項11】上記複合硬質膜は、上記基材の表面に対し、垂直方向に柱状でなる柱状結晶が含まれている請求項1~10のいずれか1項に記載の複合硬質膜被覆部材。

【請求項12】上記複合硬質膜は、該複合硬質膜中のTiとAlとの金属元素の合計含有量に対するAl元素の含有量が上記基材の表面から該複合硬質膜の表面に向かって増加している請求項1~11のいずれか1項に記載の複合硬質膜被覆部材。

【請求項13】上記複合硬質膜は、該複合硬質膜の表面粗さがJIS規格のRaによる平均表面粗さで0.1μm以下からなる請求項1~12のいずれか1項に記載の複合硬質膜被覆部材。

【請求項14】上記複合硬質膜と上記基材との間に、Tiおよび/またはAlの金属、TiとAlの合金、Tiおよび/またはAlを含む金属間化合物でなる薄層が1μm以下の膜厚さで介在されている請求項1~13のいずれか1項に記載の複合硬質膜被覆部材。

【請求項15】上記請求項1~14のいずれか1項に記載の複合硬質膜被覆部材は、切削工具として用いられる複合硬質膜被覆部材。

【請求項16】上記切削工具は、上記複合硬質膜の膜厚さが稜線部に向かって減少している請求項15に記載の複合硬質膜被覆部材。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、金属、合金、焼結合金、セラミックス焼結体または超高温高压焼結体の基材上にチタンーアルミニウム含有複合化合物でなる複合硬質膜を含む被覆層が被覆された複合硬質膜被覆部材に

関するものである。

【0002】

【従来の技術】従来から金属、合金、焼結合金、セラミックス焼結体または超高温高压焼結体の基材上に、化学蒸着法（以下、「CVD法」という）、物理蒸着法（以下、「PVD法」という）またはプラズマCVD法を利用して硬質膜を被覆し、基材と硬質膜とを有効に利用した硬質膜被覆部材が実用されてきている。現在、実用されている硬質膜被覆部材における硬質膜の材質は、Tiの窒化物、炭窒化物、炭化物などのTi元素含有硬質膜と、TiとAlを含有の複合窒化物、複合炭窒化物などのTi-Al元素含有複合硬質膜と、酸化アルミニウム硬質膜を代表例として挙げることができる。

【0003】これらの硬質膜被覆部材のうち、基材上に、Ti-Al元素含有複合硬質膜を被覆し、Ti-Al元素含有複合硬質膜の特性を有効に引き出して、長寿命を達成しようとした複合硬質膜被覆部材が多数提案されている。これらのうち、複合硬質膜の結晶構造から長寿命を達成させることについて提案されている代表的なものに、特開平8-209335号公報、特開平291353号公報、特開平9-295204号公報、特開平9-300105号公報、特開平9-300106号公報、特開平9-323204号公報、特開平9-323205号公報、特開平10-76407号公報、特開平10-76408号公報、特開平11-1762号公報、特開平11-131214号公報、特開平11-131215号公報、特開平11-131216号公報、および特開平11-131217号公報がある。また、Ti-Al元素含有複合硬質膜ではなく、Ti元素含有化合物硬質膜を被覆した被覆超硬合金について提案されている代表的なものに、特開昭52-28478号公報がある。

【0004】

【発明が解決しようとする課題】Ti-Al元素含有複合硬質膜を被覆した複合硬質膜被覆部材に関する先行技術文献のうち、特開平8-209335号公報、特開平9-295204号公報、特開平9-300105号公報、特開平9-300106号公報、特開平9-323204号公報、特開平9-323205号公報、特開平10-76407号公報、特開平10-76408号公報、特開平11-131215号公報、および特開平11-131217号公報には、Ti-Al元素含有複合硬質膜のX線回折における(111)結晶面のピーク高さに対する(200)結晶面のピーク高さの比が1以上、1.5以上または2以上である構成要件を含む被覆部材について開示されている。また、先行技術文献のうち、特開平9-291353号公報、特開平11-131216号公報には、Ti-Al元素含有複合硬質膜のX線回折における(111)結晶面のピーク高さに対する(200)結

晶面のピーク高さの比が2以下である構成要件を含む被覆部材について開示されている。

【0005】また、特開平10-317123号公報には、Cu, K α 線を線源とするX線回折における(Ti, Al)Nに代表される複合硬質膜の回折ピークのうち、(200)結晶面が最高回折ピーク高さとなる構成要件を含む複合硬質膜部材について、さらに特開平11-1762号公報には、Cu, K α 線を線源とするX線回折における同複合硬質膜の回折ピークのうち、42.5~44.5度内の回折角(2 θ)に最高回折ピーク高さを構成要件とする複合硬質膜被覆部材について開示されている。

【0006】これら15件の公開特許公報には、Ti-Al元素含有複合硬質膜における複合硬質膜内の残留圧縮応力、または複合硬質膜内の結晶配向を考慮し、複合硬質膜内の粒界破壊の抑制、基材と複合硬質膜との密着性の向上、耐摩耗性の向上を発揮させることにより、安定した切削加工と長寿命を可能としたことが開示されている。しかしながら、これら15件の同公報に記載されている複合硬質膜被覆部材は、複合硬質膜に存在する結晶の欠陥および歪みに配慮されていないことから、複合硬質膜自体の強度、耐摩耗性に満足できなく、その結果基材と複合硬質膜との密着性および複合硬質膜と隣接する他の膜との密着性に満足できなく、寿命のバラツキが大きく、切削工具として実用したときに低温領域から高温領域まで広範囲の領域において、安定して長寿命を得ることが困難であるという問題を有している。

【0007】その他、Ti-Al元素含有複合硬質膜に関する先行技術文献ではないが、特開昭52-28478号公報には、硬質膜の(200)結晶面におけるX線回折線が2 θ で半価幅が0.4度以上ある硬質膜部材について開示されている。同公報に開示の硬質膜部材は、PVD法による硬質膜であり、CVD法による硬質膜との相違を半価幅により表現しており、硬質膜の結晶の欠陥および歪みを配慮していないことから、上述した複合硬質膜と同様に硬質膜の強度、耐摩耗性および密着性に満足できなく、寿命のバラツキが大きく、切削工具として実用したときに低温領域から高温領域まで広範囲の領域において、安定して長寿命を得ることが困難であるという問題を有している。

【0008】本発明は、上述のような問題点を解決したもので、具体的には、チタンーアルミニウムを含む複合化合物でなる複合硬質膜の結晶の欠陥、歪み、結晶構造および結晶配向を配慮し、特に切削工具としての使用領域を拡大し、複合硬質膜の特性のバラツキを抑制し、高韌性、高硬度性、耐摩耗性、耐酸化性、耐熱衝撃性、耐欠損性、耐溶着性のある複合硬質膜、特に高強度および耐剥離性を高めて被削材との耐溶着性を向上させた複合硬質膜とすることにより一層長寿命を達成させた複合硬質膜被覆部材の提供を目的とするものである。

【0009】

【課題を解決するための手段】本発明者らは、CVD法、PVD法およびプラズマPVD法に関する硬質膜の成膜についての研究、特にPVD法による硬質膜についての研究を長期に亘って行ってきた結果、硬質膜の中でもTi-Al含有の複合硬質膜の成膜時におけるプラズマ密度の向上およびイオン化効率の向上を行い、さらに気相法エピタキシャル成長させて、結晶を最適に配向させると、複合硬質膜内の歪みが均一に緩和されること、複合硬質膜の結晶の欠陥が抑制されること、微細結晶の複合硬質膜が得られることから、複合硬質膜自体の強度、耐摩耗性、耐酸化性および耐熱性を向上させることができとなり、複合硬質膜と基材または下地層や外層との密着性の向上が顕著になるという第1の知見と、このように硬質膜が完全な結晶に近似する場合には、X線回折における最高ピークを含めた少なくとも2本の回折線ピークの高さ比および半価幅比から判断することが簡易であるという第2の知見を得るに至ったものである。これらの知見に基づいて、本発明を完成するに至ったものである。

【0010】本発明の複合硬質膜被覆部材は、基材の表面にチタンとアルミニウムとを含む複合窒化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さをh(200)とし、(111)結晶面のピーク高さをh(111)としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅をd(200)とし、該(111)結晶面のピークの半価幅をd(111)としたときに、 $1.5 \geq d(200)/d(111) \geq 0.8$ からなるものである。

【0011】本発明の複合硬質膜被覆部材は、気相法エピタキシャル成長を利用して、チタンとアルミニウムとを含む複合硬質膜における(200)結晶面の配向を強くし、複合硬質膜内の歪みを極力抑制することにより、複合硬質膜自体の強度、韌性を高めると共に、耐摩耗性もすぐれるというシナージ効果を発揮させたものであり、複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、 $h(200)/h(111) < 4.0$ になる場合、 $d(200)/d(111) > 1.5$ または $d(200)/d(111) < 0.8$ になる場合には、(200)結晶面への配向性が弱く、膜内の欠陥および歪みが大きくなり、上述のシナージ効果が弱くなることから、上述のようなピーク高さ比および半価幅比を定めたものである。

【0012】

【発明の実施の形態】本発明の複合硬質膜被覆部材における基材は、被覆層を被覆するときに加熱する温度に耐

えることができる材料または物質ならば使用することができる、具体的には、例えばステンレス鋼、耐熱合金、高速度鋼、ダイス鋼、Ti合金、Al合金に代表される金属部材、超硬合金、サーメット、粉末ハイスに代表される焼結合金、Al₂O₃系焼結体、Si₃N₄系焼結体、サイアロン系焼結体、ZrO₂系焼結体、炭化珪素系焼結体に代表されるセラミックス焼結体、立方晶窒化硼素系焼結体、ダイヤモンド系焼結体に代表される超高温高圧焼結体を挙げることができる。これらのうち、従来から切削用工具または耐摩耗用工具として用いられている材料または物質を基材とする場合には、被覆切削用工具または被覆耐摩耗用工具としての寿命向上の効果が高くなることから、好ましいことである。

【0013】これらの基材のうち、超硬合金を基材とする場合には、主成分がCoおよび/またはNiでなる結合相を3~15重量%と、残部の炭化タングステンまたは炭化タングステンと周期律表の4a、5a、6a族金属の炭化物、炭窒化物およびこれらの相互固溶体から選ばれた少なくとも1種の硬質相とを含有する超硬合金にすると、基材の強度、韌性および耐摩耗性などの特性と複合硬質膜の特性とを最適に發揮させることができとなり、切削工具としての寿命向上効果が顕著となることから、特に好ましいことである。このときの超硬合金を構成している結合相および硬質相は、従来の超硬合金に含有されている結合相および硬質相の組成成分からなるものを実用できる。

【0014】また、セラミックス焼結体を基材にする場合には、窒化珪素および/またはサイアロンを80~98重量%と、残部の焼結助剤からなる窒化珪素系焼結体を基材とすると、基材の強度、韌性、耐摩耗性、熱伝導性などの機械的特性および物理的特性と複合硬質膜の機械的特性および物理的特性から、基材と複合硬質膜との整合性を最適に發揮させることができとなり、切削工具としての寿命向上効果が顕著となり、特に好ましいことである。このときの窒化珪素系焼結体を構成している窒化珪素が α 窒化珪素および/または β 窒化珪素でなる場合、サイアロンが α サイアロンおよび/または β サイアロンでなる場合でもよく、焼結助剤が従来から窒化珪素系焼結体やサイアロン系焼結体に含有されている物質を用いることができるものであり、具体的な焼結助剤としては、例えばY、La、Ce、Dy、などの希土類元素の酸化物、酸化マグネシウム、酸化ハフニウム、酸化ジルコニウム、酸化アルミニウム、窒化アルミニウム、酸化珪素およびこれらの相互固溶体から選ばれた少なくとも1種の焼結助剤を挙げができる。

【0015】さらに、ダイヤモンド系焼結体、立方晶窒化硼素系焼結体に代表される超高温高圧焼結体を基材にする場合には、70体積%以上のダイヤモンドと残部の粒界相とからなるダイヤモンド系焼結体、ならびに20体積%以上の立方晶窒化硼素と粒界結合相とからなる立

方晶窒化硼素系焼結体となる場合が好ましいことである。これらのうち、ダイヤモンド系焼結体は、ダイヤモンド（以下、「DIA」と記す）が85～98体積%と残部の粒界相が従来のダイヤモンド系焼結体に含有されている金属、合金、具体的には、Co, Ni, Fe, Siおよびこれらの相互固溶体の中の少なくとも1種を含む場合には、基材と複合硬質膜との両特性を最適に発揮させることができ、切削工具としての寿命向上が顕著になることから好ましいことである。立方晶窒化硼素系焼結体は、立方晶窒化硼素が35～95体積%と残部の粒界結合相が周期律表の4a, 5a, 6a族元素の炭化物、窒化物、硼化物、Si, Mg, Alの窒化物、硼化物、酸化物およびこれらの相互固溶体、Co, Ni, Ti, Alの金属、合金、金属間化合物の中から選ばれた少なくとも1種からなる場合には、基材と複合硬質膜との両特性を最適に発揮させることができ、切削工具としての寿命向上が顕著になることから好ましいことである。

【0016】これらの基材に共通した問題として、基材の表面精度があり、基材の表面精度を高くすると、複合硬質膜の表面精度も高くなり、例えば、切削工具として使用した場合に摩擦抵抗が低くなつて複合硬質膜表面および被削材表面の荒れが抑制されて、寿命向上効果が高くなることから好ましいことである。基材の表面精度は、JIS規格B0601に規定されている表面粗さにおける中心線平均粗さであるRaで0.1μm以下が好ましく、より好ましいのはRaが0.05μm以下からなるものである。

【0017】これらの基材表面に被覆される複合硬質膜を含む被覆層の構成は、基材に隣接して密着性を目的に被覆される下地層、この下地層に隣接して被覆される本発明における複合硬質膜である中間層、この中間層に隣接して被覆される外層、この外層の表面に使用前後の判別および装飾目的で被覆される最外層などを2層以上に積層する構成、具体的には、例えば基材の表面に順次被覆される被覆層が基材一下地層一複合硬質膜（中間層）一外層一最外層からなる積層の構成、基材一下地層一複合硬質膜（中間層）一外層からなる積層の構成、基材一下地層一複合硬質膜（中間層）の積層からなる構成、基材一下地層一複合硬質膜（中間層）一最外層からなる積層の構成、基材一複合硬質膜（中間層）一外層一最外層からなる積層の構成、基材一複合硬質膜（中間層）一外層からなる積層の構成、基材一複合硬質膜（中間層）一最外層の積層からなる構成、または基材一複合硬質膜（中間層）の構成、を挙げることができる。これらのうち、基材表面に直接に複合硬質膜を被覆する構成の場合には、製造時における工程の煩雑さがないこと、工程時間の短縮となること、品質管理上のバラツキが少なくなることから、好ましいことである。

【0018】これらの被覆層のうち、下地層は、金属、

10

20

30

40

50

合金、金属間化合物または金属化合物であり、具体的には、例えばTi, Al, Ni, Co, Wの金属、これらの相互合金、Ti-Al, Ti-Ni, Ti-Co, Al-Ni, Al-Co, Co-W, Ti-Al-Ni, Ti-Al-Coの金属間化合物、周期律表の4a, 5a, 6a族金属の炭化物、窒化物、炭酸化物、窒酸化物、これらの相互固溶体の金属化合物から選ばれた少なくとも1種の单層または多層でなる場合を挙げができる。また、外層は、具体的には、例えば周期律表の4a, 5a, 6a族金属の炭化物、窒化物、炭酸化物、窒酸化物、これらの相互固溶体、Alの酸化物、窒化物、酸窒化物、ダイヤモンド、硬質カーボン（ダイヤモンド状カーボンともいわれる）、立方晶窒化硼素、硬質窒化硼素、これらの2種以上の混合物の中から選ばれた少なくとも1種の单層または多層でなる場合を挙げができる。さらに、最外層は、使用前後の判別が容易な色彩を有する被覆層、装飾的効果のある被覆層であればよく、具体的には、例えば4a, 5a, 6a族金属の窒化物、炭窒化物、窒酸化物、これらの相互固溶体の中から選ばれた少なくとも1種の单層または多層でなる場合を挙げができる。

【0019】本発明の骨子となる複合硬質膜は、被覆層自体が複合硬質膜である場合、別の表現をすると、基材表面に複合硬質膜のみが被覆された構成である場合、基材表面に上述の下地層と複合硬質膜とが被覆されている構成の場合、または前述の被覆層の構成のように中間層として被覆されている構成の場合がある。この複合硬質膜の組成成分、膜質は、具体的な例示として化学式により記載すると、(Ti, Al)N、(Ti, Al)C、(Ti, Al)(C, N)、(Ti, Al)(N, O)、(Ti, Al)(C, O)、(Ti, Al)(C, N, O)、(Ti, Al, M)N、(Ti, Al, M)C、(Ti, Al, M)(C, N)、(Ti, Al, M)(N, O)、(Ti, Al, M)(C, O)、および(Ti, Al, M)(C, N, O)の中から選ばれた少なくとも1種の单層または積層でなる場合を挙げができる。（ただし、Mは、Ti, Alを除いた金属および半金属の元素の1種以上を表わし、特に周期律表の4a, 5a, 6a族元素、希土類元素、Mn元素、Mg元素、Si元素、B元素の中の少なくとも1種からなる場合が好ましい）

【0020】これらの複合硬質膜は、金属元素がTiとAlのみを含有している場合には、次の化学式で表せる $(Ti_a, Al_b)(C_x, N_y, O_z)_w$ の複合硬質膜【ただし、aは金属元素中のTi（チタン）元素の原子比、bは金属元素中のAl（アルミニウム）元素の原子比、xは非金属元素中の炭素（C）元素の原子比、yは非金属元素中の窒素（N）元素の原子比、zは非金属元素中の酸素（O）元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれがa+b=1、

$0.8 \geq a \geq 0.4$ 、 $x+y+z=1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]でなる場合には、複合硬質膜自体の強度、耐摩耗性および韌性にすぐれること、しかも歪み、欠陥が少なく耐剥離性にすぐれることから好ましいことである。

【0021】また、TiとAl以外の金属元素を含有した複合硬質膜でなる場合には、次の化学式で表せる (T_{ia} , A_{1b} , M_{1-a-b}) (C_x , N_y , O_z)_w の複合硬質膜 [ただし、aは金属元素中のTi (チタン) 元素の原子比、bは金属元素中のAl (アルミニウム) 元素の原子比、Mは周期律表の4a, 5a, 6a族元素、希土類元素、Si元素、Mn元素、Mg元素、B元素の中の少なくとも1種を表し、xは非金属元素中の炭素 (C) 元素の原子比、yは非金属元素中の窒素 (N) 元素の原子比、zは非金属元素中の酸素 (O) 元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれが $0.8 \geq a \geq 0.4$ 、 $0.6 > b > 0.2$ 、 $x + y + z = 1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]でなる場合には、複合硬質膜自体の強度、耐摩耗性および韌性にすぐれること、しかも歪み、欠陥が少なく耐剥離性にすぐれることから好ましいことである。

【0022】これらの複合硬質膜は、複合硬質膜の結晶粒子の界面に金属元素からなる複合硬質膜強化物質が微量に含有されると、より一層複合硬質膜自体の強度、韌性がすぐれること、歪みが緩和されること、耐剥離性の向上が顕著になることから好ましいことである。このときの複合硬質膜強化物質は、基材を構成している金属元素からなる場合には、基材と複合硬質膜との整合性を高めること、密着性を高めることから好ましいことである。この複合硬質膜強化物質は、被覆層を被覆する前に、メッキ法や真空蒸着法などにより形成しておいて、これを拡散させることも可能であるが、基材を構成している金属元素を複合硬質膜中に拡散させると簡易に得られることから、好ましいことである。

【0023】これらの複合硬質膜は、複合硬質膜中のTiとAlとの金属元素の合計含有量に対するAl元素の含有量が基材表面から複合硬質膜の表面に向かって増加していること、別の表現をすると、Ti元素の含有量が複合硬質膜の表面から基材表面に向かって増加していること、いわゆる傾斜組成の複合硬質膜にすると基材と複合硬質膜との密着性がすぐれること、複合硬質膜自体の強度、韌性にすぐれて、欠陥、歪みおよび残留応力が減少すること、複合硬質膜表面の耐酸化性、耐摩耗性および耐腐食性がすぐれることから、好ましいことである。このときのAl元素およびTi元素の増加は、階段状、ノコギリの刃状にミクロ的には増減があるとしてもマクロ的には段階的に増加する場合、放物線状、直線状に連続的に増加している場合でもよいものである。

【0024】この複合硬質膜は、複合硬質膜自体の構造からすると、基材表面に対し垂直方向に柱状に成長した柱状結晶が含まれている場合には、複合硬質膜の表面からの耐圧壊強度が向上し、耐剥離性、耐微小チッピング性にすぐれることから、好ましいことである。この柱状結晶を含む複合硬質膜は、具体的には、複合硬質膜全体が柱状結晶の層でなる場合、粒状結晶と柱状結晶との混在した層でなる場合、粒状結晶の層と柱状結晶の層との積層でなる場合、またはこれらの粒状結晶と柱状結晶のそれぞれの中に前述した複合硬質膜強化物質が微量含有されている場合を例示することができる。これらのうち、複合硬質膜強化物質が複合硬質膜と複合硬質膜強化物質との合計に対し、3体積%以下、好ましくは1体積%以下含有していると、複合硬質膜の表面からの垂直方向および水平方向の両方からの耐圧壊強度、耐圧縮強度にすぐれるとともに、耐摩耗性にもすぐれるというシナジー効果を発揮することができることから、好ましいことである。

【0025】これらの複合硬質膜は、前述した被覆層の構成のどの位置に存在するかにより複合硬質膜自体の構造を配慮することが好ましく、この被覆層の構成として、基材に直接複合硬質膜を被覆する場合、または基材に下地層を被覆した後、下地層に複合硬質膜を被覆し、複合硬質膜の表面が他の物質（例えば、切削工具における被削材）と接触する状態で使用される第1の構成による複合硬質膜部材と、複合硬質膜の表面に外層を被覆した場合、または複合硬質膜の表面に外層および最外層を被覆し、複合硬質膜の表面が他の物質と直接接触しない状態で使用される第2の構成による複合硬質膜部材とに大別することができる。

【0026】これらのうち、第1の構成による複合硬質膜部材の場合には、複合硬質膜の表面は、JIS規格B 0601に規定されている表面粗さにおける中心線平均粗さRaで $0.1 \mu\text{m}$ 以下、好ましくは $0.05 \mu\text{m}$ 以下にすると、切削工具として使用した場合に、被削材への損傷が緩和されること、切削抵抗が緩和されること、切粉の排出が容易になることから、より一層の長寿命となり、好ましいことである。また、第2の構成による複合硬質膜部材においても、外層の膜厚さ、または外層と最外層との合計膜厚さにより異なるが、複合硬質膜の表面粗さを上述のようにしておくと、外層および最外層の表面粗さも滑らかで、平坦となり、上述と同様の効果を発揮できることから、好ましいことである。

【0027】被覆層を構成する各層の膜厚さは、用途、形状および被覆層の構成により、選択されるのであるが、上述の第1の構成による複合硬質膜部材の場合には、被覆層の主体が複合硬質膜となり、この場合には複合硬質膜の膜厚さを $1 \sim 20 \mu\text{m}$ 、主として密着性を目的として下地層を介在させる場合には、下地層の膜厚さを $0.2 \sim 2 \mu\text{m}$ とすると、被覆層自体の強度、耐摩耗

11

性、韌性および耐剥離性から、好ましいことである。また、上述の第2の構成による複合硬質膜部材でなる場合には、下地層の膜厚さを0.2~2μm、複合硬質膜の膜厚さを1~10μm、外層の膜厚さを1~10μm、最外層の膜厚さを0.5~2μmとすると、各膜層の特性を最適に発揮させることができることから、好ましいことである。以上に詳述してきた複合硬質膜を初め、下地層、外層、および最外層は、化学量論組成でなる場合、または非化学量論組成でなる場合でもよく、実質的には非化学量論組成からなっている場合が多いものである。

【0028】以上のような形態でなる本発明の複合硬質膜部材は、各種の用途に実用できるものであり、具体的には、例えば旋削工具、フライス工具、ドリル、エンドミルに代表される切削工具、特に被削材が鉄物や鋼であり、耐衝撃性を必要とする断続切削工具や回転切削工具として、ダイス、パンチなどの型工具からスリッターなどの切断刃、裁断刃などの耐摩耗用工具として、ノズルや塗付工具などの耐腐食耐摩耗用工具として、鉱山、道路、土建などに用いられる切断工具、掘削工具、锪孔工具、破碎工具に代表される土木建設用工具として実用できるものである。これらのうち、本発明の複合硬質膜部材は、ミクロ的に温度、摩擦、熱衝撃および圧縮衝撃などが最も過酷な条件となる切削工具、特にドリル、エンドミルなどの回転切削工具、スローアウエイチップなどの切削工具として使用する場合には、複合硬質膜の特性を最適に発揮させ得ることから、好ましいことである。この複合硬質膜部材を切削工具として使用する場合には、複合硬質膜の膜厚さは、切削工具の切刃に形成される稜線部に向かって減少するように形成すると、耐剥離性、微小チッピング性にすぐれることから好ましいことである。また、これらの複合硬質膜を含む被覆層の膜厚さが切削工具の切刃に形成される稜線部に向かって減少するように形成されることも、同様の効果を惹起させることになり、好ましいことである。

【0029】この本発明の複合硬質膜被覆部材は、従来から市販されているステンレス鋼、耐熱合金、高速度鋼、ダイス鋼、Ti合金、Al合金に代表される金属部材、超硬合金、サーメット、粉末ハイスに代表される焼結合金、Al₂O₃系焼結体、Si₃N₄系焼結体、サイアロン系焼結体、ZrO₂系焼結体、炭化珪素系焼結体に代表されるセラミックス焼結体、立方晶窒化硼素系焼結体、ダイヤモンド系焼結体に代表される超高温高圧焼結体を基材とし、この基材の表面を、必要に応じて研磨し、超音波洗浄、有機溶剤洗浄などを行った後に、従来から行われているPVD法、CVD法またはプラズマCVD法により基材上に被覆層を被覆して作製することができるが、以下の方法で作製すると、プラズマ密度の向上とイオン化効率の向上が可能となること、複合硬質膜自体の気相エピタキシャル結晶成長および結晶配向が容

12

易となること、複合硬質膜の特性および密着性がよりすぐれることから、好ましいことである。

- 【0030】この複合硬質膜被覆部材を得るための製造方法として、重要な特徴について具体的に詳述すると、基材の表面は、従来から行われているプラスト処理、ショットビーニング処理、研磨処理、バレル処理の中の少なくとも1種の機械的処理と、酸性もしくはアルカリ性の電解液による電解エッチング、酸溶液、アルカリ溶液による表面腐食、または水、有機溶液による洗浄の中の10少なくとも1種の化学的処理と、この機械的処理と化学的処理を同時または別々に行う処理方法とから選択される処理を行うと、基材表面の欠陥を除去できること、複合硬質膜の密着性を高め得ること、膜内歪みを抑制できること、膜内の欠陥を抑制できることから、好ましいことである。また、基材は、このような機械的処理および/または化学的処理と、低温による熱処理を付加して、上述の効果を高めることも好ましいことである。
- 【0031】基材の表面に複合硬質膜を被覆する場合は、スパッター法やイオンプレーティング法に代表されるPVD法により行なうことが好ましく、これらのうち、マグネトロンスパッター法またはアークプラズマイオンプレーティング法により行なうと、複合硬質膜の調整が容易であることから、特に好ましいことである。具体的には、例えばイオンプレーティング装置の反応容器内に基材を配置し、基材表面をポンバード処理する場合に、金属元素イオンによるポンバード処理、もしくは金属元素イオンと非金属元素イオンとの両方によるポンバード処理を施すと、上述の効果を高めることになることから、好ましいことである。特に、前述の下地層のうち、金属、合金または金属間化合物の下地層を必要とする場合は、金属元素イオンを含むイオンポンバードを施すと、下地層の形成が容易であること、基材と下地層との密着性が高くなることから、好ましいことである。
- 【0032】複合硬質膜の被覆条件は、反応容器の構造、プラズマの調整など装置自体の影響を重要視する必要があり、具体的には、例えば高電圧、(場合によってはパルス状高電圧と高周波を付加)の電源でイオンを加速とプラズマ発生させる装置、磁界によるプラズマの調整可能な装置を使用すること、その他、反応容器内の雰囲気圧力、温度、アーク放電電流、電圧、基材バイアス電圧、試料の配置などについて配慮する必要があり、これらのうち、従来の条件に対し、特にアーク放電電圧を高くすること、基材バイアス電圧を高くすること、試料の回転および上下動などが重要な要件である。

【0033】

【実施試験1】以上に詳述してきた本発明の実施形態について、さらに具体的な代表例として実施試験により説明する。まず、従来の配合、混合、成形、焼結の各工程を経て作製された超硬合金の基材とサーメットの基材を用いて、これらの基材表面に直接複合硬質膜を被覆した

試験について説明する。基材は、配合組成成分で示した表1により作製されたISO規格によるSNGN120408形状の超硬合金の基材1～基材5とサーメットの基材6を用い、これらの基材の上下面と外周面を270#のダイヤモンド砥石で研削加工を施し、刃先部に400#ダイヤモンド砥石により $-25^\circ \times 0.10\text{ mm}$ のホーニング加工を施し、さらに表面を湿式ブلاスト処理、洗浄処理および乾燥処理を行った後、アークイオンプレーティング装置により複合硬質膜を被覆した。

【0034】処理条件は、反応容器内の各基材表面をポンバード処理した後、複合硬質膜を被覆した。ポンバード処理は、反応容器内の雰囲気：真空、基材温度：873K、アーク電流：70A、基材バイアス電圧：-600V、Arガスポンバードにより行った。複合硬質膜の被覆は、反応容器内のガス流量：200～350SCCM、蒸発源：Ti-Al合金、アーク電圧：200～300V、アーク電流：150～200A、基材温度：773～873K、基材バイアス電圧：-100～-200Vにより行い、表1に示した基材1～6のそれぞれの表面に複合硬質膜を被覆して表2に示した本発明品1～6を得た。これらのうち、反応容器内の雰囲気は、本発明品1がAr-N₂-O₂ガス組成、本発明品2がAr-N₂-COガス組成、その他本発明品3～6がAr-N₂ガス組成で行い、本発明品4の蒸発源には、Ti元素の多い合金からAl元素の多い合金に切り替えて行い、本発明品5の蒸発源には、Ti元素の多い合金-Al元素の多い合金-Ti元素の多い合金に切り替えて行った。

【0035】比較として、表1に示した基材3～6のそれぞれの表面に複合硬質膜を被覆して表2に示した比較品1～4を得た。基材表面粗さは、上述の本発明品1～6に使用の基材表面がRa=0.01～0.005μmに対し、比較品1～4に使用の基材表面がRa=0.1～0.05μmであった。比較品1～4における基材処理は、上述した本発明品の基材表面処理のうち、湿式ブلاスト処理を除いて、その他はほぼ同様に処理した。また、同比較品1～4における複合硬質膜の被覆は、上述した本発明品の複合硬質膜の処理条件のうち、アーク電圧：10～50V、アーク電流：200～250A、基材

10

1～4のそれぞれの複合硬質膜について、X線回折装置、走査型電子顕微鏡、金属顕微鏡、EDS装置、ピッカース硬さ試験機および引っ掻き硬さ試験機に相当するスクラッチ試験機を用いて、複合硬質膜表面からのX線回折によるh(200)/h(111)、d(200)/d(111)、複合硬質膜表面のTiとAlの含有率、複合硬質膜表面の硬さ、スクラッチ強度を求めて、それぞれの結果を表2に示した。なお、本発明品1～6および比較品1～4の複合硬質膜厚さは、ほぼ5～7μmからなり、本発明品1～6の複合硬質膜表面の粗さは、ほぼRa=0.015～0.010μmであり、比較品1～4の複合硬質膜表面の粗さは、ほぼRa=0.15～0.10μmであった。また、本発明品1の複合硬質膜は、非金属元素中の酸素元素が1at%以下の窒化膜(Ti, Al)(N, O)であり、本発明品2の複合硬質膜は、非金属元素中の酸素元素が2at%以下の炭窒化膜(Ti, Al)(N, C)であり、その他の複合硬質膜は、(Ti, Al)Nで表示される窒化膜であった。

20

【0036】次いで、本発明品1～5および比較品1～3を用いて、以下の切削条件により湿式断続切削試験を行った。切削条件は、被削材：機械構造用炭素鋼材のS45Cに4本の溝入丸棒、切削速度：150m/mi n、送り：0.3mm/rev、切り込み：2.0mm、工具形状：SNGN120408、水溶性切削油により行った。湿式断続切削試験の結果は、切刃のチッピング、被覆層の剥離したとき、平均逃げ面摩耗量または境界摩耗量が0.3mmに達したときを工具寿命とし、そのときのそれぞれの切削可能時間を求めて、比較品1の寿命に対する寿命比として、表2に併記した。

30

【0037】

【表1】

試料番号	焼結合金の組成成分(配合時)	重量%
基材1	97WC-3Co	
基材2	91WC-3TaC-1TiC-5Co	
基材3	88WC-2TaC-2TiC-8Co	
基材4	86.5WC-1TaC-0.5NbC-2TiC-10Co	
基材5	80WC-1Cr3C2-1VC-18Co	
基材6	26TiC-26TiN-20WC-10TaC-1Mo2C-1ZrC-8Ni-8Co	

【0039】

※※【表2】

15

16

試料番号	複合硬質膜の結晶面ピーク		複合硬質膜の成分比、特性			切削試験 寿命比	
	高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)		
本発明品	1	5.0	0.85	52:48	2780	70	2.5
	2	4.6	0.9	55:45	2950	75	2.8
	3	7.5	1.1	53:47	2900	90	5.2
	4	6.3	1.4	56:44	2920	86	4.8
	5	5.8	1.2	58:42	3000	80	3.5
	6	6.5	1.3	50:50	2880	83	なし
比較品	1	3.7	1.2	53:47	2910	43	1.0
	2	1.4	2.3	50:50	2950	34	0.9
	3	2.4	2.5	55:45	2850	40	1.1
	4	0.1	0.6	40:60	3100	25	なし

【0040】

【実施試験2】実施試験1の表1に示した基材3の超硬合金を基材とし、アーキオイオンプレーティング装置を用いて、この基材の表面に下地層、複合硬質膜、外層、最外層の中の2層以上を含む積層の被覆層を被覆した本発明品7～13と比較品5と比較品6を得た。これらのうち、本発明品7のTi下地層は、基材表面にTi蒸着をし、本発明品8の(Ti-Al)金属間化合物下地層は、(Ti-Al)蒸発源によるポンバード処理と同時に蒸着し、本発明品9のTi下地層は、Ti蒸発源によるポンバード処理と同時に蒸着した。本発明品7～13の基材処理および複合硬質膜の成膜は、実施試験1における本発明品3～6とほぼ同様に処理し、比較品5および比較品6の基材処理および複合硬質膜の成膜は、実施試験1における比較品1とほぼ同様に処理した。その他の下地層、外層および最外層は、ほぼ従来の製法により処理した。

【0041】こうして得た本発明品7～13と比較品5および比較品6のそれぞれの下地層、複合硬質膜、外層*

*および最外層について、実施試験1と同様にして調べて、それぞれの被覆層の構成と各膜質および膜厚さを表3に示し、複合硬質膜表面におけるX線回折による結晶面ピーク高さ比、半価幅比、TiとAlの元素比、表面硬さ、スクラッチ強度を表4に示した。また、本発明品7～13と比較品5および比較品6について、実施試験1の切削条件と同様にして切削試験を行い、比較品5に対するそれぞれの寿命比を求めて、表4に併記した。なお、複合硬質膜の表面調査は、複合硬質膜を被覆した後に反応容器から取り出して行い、本発明品10は、全被覆終了後に、熱処理を行って基材中の結合相を被覆層中に拡散させ、下地層および複合硬質膜に約1体積%の結合相を含有させた。また、本発明品11は、下地層の炭窒化チタン層が柱状晶からなり、かつ複合硬質膜の成膜時にプラズマ密度、ラジカル反応および成膜速度を配慮して柱状晶結晶としたものである。

30 【0042】

【表3】

試料番号	被覆層の膜質および膜厚さ(μm)			
	下地層	複合硬質膜	外層	最外層
本発明品	7 0.5Ti	6(Ti,Al)N	なし	なし
	8 0.5(Ti-Al)	6(Ti,Al)N	なし	1TiN
	9 0.5Ti-2TiN	6(Ti,Al)N	なし	なし
	10 1TiN	3(Ti,Al)N	5Al ₂ O ₃	なし
	11 1TiN-3TiCN	3(Ti,Al)N	5Al ₂ O ₃	1TiN
	12 1.5TiN	3(Ti,Al)N	2TiCO-3Al ₂ O ₃	1TiCN
	13 1.5TiN	4(Ti,Al)N	2TiCO-3Al ₂ O ₃	1TiN
比較品5	1TiN	3(Ti,Al)N	5Al ₂ O ₃	なし
比較品6	1.5TiN	4(Ti,Al)N	2TiCO-3Al ₂ O ₃	1TiN

【0043】

※ ※【表4】

17

18

試料番号	複合硬質膜の結晶面ピーク		複合硬質膜の成分比、特性			切削試験 寿命比	
	高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)		
本発明品	7	7.3	1.2	58:42	2900	65	5.7
	8	7.4	1.3	53:47	2950	62	4.8
	9	7.6	1.2	55:45	2900	76	5.5
	10	7.8	1.1	54:46	2910	96	7.1
	11	8.5	1.0	50:50	3000	88	6.5
	12	7.4	0.9	51:49	2890	73	5.1
	13	7.2	0.95	57:43	2920	70	4.7
比較品6	3.3	3.0	54:46	2900	35	1.0	
比較品6	3.0	2.4	53:47	2930	42	1.3	

【0044】

【実施試験3】70体積%Al₂O₃-30体積%TiC N(配合組成)により作製されたセラミックス焼結体の基材7と、70体積%Al₂O₃-30体積%SiCウイスカ- (配合組成)により作製されたセラミックス焼結体の基材8と、96体積%Si₃N₄-1体積%MgO-1体積%Y₂O₃-2体積%HfO₂ (配合組成)により作製されたセラミックス焼結体の基材9と、90体積%Si₃N₄-4体積%AlN-4体積%Al₂O₃-1体積%MgO-1体積%Y₂O₃ (配合組成)により作製されたセラミックス焼結体の基材10を用いて、実施試験1における本発明品3~6とほぼ同様に基材を処理し、各基材表面に約1μm膜厚さのTiを蒸着した後に、複合硬質膜を被覆し、本発明品14~17を得た。また、比較として、基材7と基材9を用いて、実施試験1における比較品1とほぼ同様に処理し、比較品7および比較品8を得た。

【0045】こうして得た本発明品14~17と比較品7及び比較品8の複合硬質膜について、実施試験1と同様にして調べて、複合硬質膜表面におけるX線回折によ*

*る結晶面ピーク高さ比、半価幅比、TiとAlの元素比、表面硬さ、スクラッチ強度を表5に示した。また、本発明品14と本発明品15と比較品7は、被削材:FCD600、切削速度:150m/min、切込み:1.5mm、送り:0.2mm/刃、工具形状:SNGN120408、ホールディング:0.15×-25°、乾式フライス切削試験を行った。本発明品16と本発明品17と比較品8は、被削材:FCD250、切削速度:600m/min、切込み:3.0mm、送り:0.1mm/刃、工具形状:SNGN120412、ホールディング:0.15×-25°、乾式フライス切削試験を行った。これらの乾式フライス結果は、実施試験1における切削試験と同様にし、本発明品14と本発明品15は、比較品7に対する寿命比とし、本発明品16と本発明品17は、比較品8に対する寿命比として表5に併記した。なお、本発明品14~17と比較品7及び比較品8の複合硬質膜厚さは、約5μmであった。

【0046】

【表5】

試料番号	複合硬質膜の結晶面ピーク		複合硬質膜の成分比、特性			切削試験 寿命比	
	高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)		
本発明品	14	6.8	0.95	60:40	2880	75	6.5
	15	6.3	1.05	62:38	2850	68	6.1
	16	12.5	1.1	48:52	3150	70	5.3
	17	13.4	1.2	45:55	3200	73	6.0
比較品7	3.5	2.3	58:42	2950	31	1.0	
比較品8	3.7	2.1	46:54	3130	32	1.0	

【0047】

【実施試験4】40体積%BN-5体積%Al₂O₃-5体積%AlN-10体積%Al-10体積%Mg-10体積%BN-20体積%TiN (配合組成)により作製された超高温高圧の立方晶窒化硼素系焼結体の基材11と、85体積%BN-2体積%C-5体積%Al-2体積%Mg-6体積%TiN (配合組成)により作製された超高温高圧の立方晶窒化硼素系焼結体の基材12※50

※と、95体積%DIA-2体積%Co-2体積%Ni-1体積ZrC (配合組成)により作製された超高温高圧のDIA系焼結体の基材13と、97体積%DIA-1体積%Co-1体積%Ni-1体積Mg (配合組成)により作製された超高温高圧のDIA系焼結体の基材14を用いて、実施試験1における本発明品3~6とほぼ同様に基材を処理し、基材11と基材12の表面に約1μm膜厚さのTiを蒸着し、基材13と基材14の表面に

19

約 $1\mu\text{m}$ 厚さのNi無電解メッキを施した後に、複合硬質膜を被覆し、本発明品18~21を得た。また、比較として、基材11と基材13を用いて、実施試験1における比較品1とはほぼ同様に処理し、比較品9および比較品10を得た。

【0048】こうして得た本発明品18~21と比較品9及び比較品10の複合硬質膜について、実施試験1と同様にして調べて、複合硬質膜表面におけるX線回折による結晶面ピーク高さ比、半価幅比、TiとAlの元素比、表面硬さ、スクラッチ強度を表6に示した。また、本発明品18と本発明品19と比較品9は、被削材：S*

20

* CM415（硬さ：約HRC61）、切削速度：150m/min、切込み：0.5mm、送り：0.1mm/rev、工具形状：Tnma160408、ホーニング：0.15×-25°、外周連続乾式旋削試験を行い、その結果を表6に併記した。この外周連続乾式旋削試験の評価は、実施試験1における切削試験と同様に行い、比較品9に対する寿命比として表した。なお、本発明品18~21と比較品9及び比較品10の複合硬質膜厚さは、約 $5\mu\text{m}$ であった。

10 【0049】

【表6】

試料番号	複合硬質膜の結晶面ピーク		複合硬質膜の成分比、特性			切削試験 寿命比	
	高さ比：h (200)/(111)	半価幅比：d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)		
本発明品	18	19.5	1.1	58:42	2960	85	8.2
	19	18.4	1.0	50:41	2930	83	8.8
	20	8.5	1.05	55:45	2950	76	なし
	21	8.9	0.95	54:48	2970	74	なし
比較品9	3.1	2.0	55:45	2910	23	1.0	
比較品10	3.5	1.7	52:48	2940	25	なし	

【0050】

【発明の効果】本発明の複合硬質膜被覆部材は、気相法エピタキシャルによる結晶成長と結晶配向による複合硬質膜が被覆されていること、複合硬質膜自体の歪み、欠陥が抑制されていること、微細結晶であること、場合によっては柱状結晶および／または微量の金属などの複合硬質膜強化物質が含まれた複合硬質膜であることから、従来の複合硬質膜被覆部材または本発明から外れた複合硬質膜被覆部材に対比して、基材と複合硬質膜、下30地層と複合硬質膜、複合硬質膜と外層など、複合硬質膜※

※に隣接する物質に対し、密着性および耐剥離性が非常にすぐれること、複合硬質膜自体の高韌性、高強度、耐熱性、耐熱衝撃性、耐酸化性および耐摩耗性がすぐれていこと、その結果として例えば切削工具として使用した場合に、切削工具として重要視される高韌性、耐摩耗性、耐熱衝撃性、耐欠損性、耐酸化性および耐溶着性が顕著に向上し、長寿命化が達成されること、切削加工における高効率化が達成されること、バラツキが小さく安定しているという顕著な効果がある。

【手続補正書】

【提出日】平成12年12月8日(2000.12.8)

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】0032

【補正方法】変更

【補正内容】

【0032】複合硬質膜の被覆条件は、反応容器の構造、プラズマの調整など装置自体の影響を重要視する必要があり、具体的には、例えば高電圧（場合によっては

パルス状高電圧と高周波を付加）の電源でイオンを加速し、プラズマを発生させる装置、さらに磁界によるプラズマの調整可能とした装置を使用すること、その他反応容器内の雰囲気圧力、温度、アーク放電電流、電圧、基材バイアス電圧、試料の配置などについて配慮する必要があり、これらのうち、従来の条件に対し、特にアーク放電電圧を高くすること、基材バイアス電圧を高くすること、試料の回転および上下動などが重要な要件である。

フロントページの続き

(51) Int.Cl. ⁷	識別記号	F I	テーマコード(参考)
B 3 2 B 15/04		B 3 2 B 15/04	

F ターム(参考) 3C046 FF03 FF04 FF10 FF25 FF32
FF33 FF35 FF39 FF53
4F100 AA12B AA15B AB31A AT00A
BA02 EH66 EH662 EJ34
EJ342 GB51 JK01 JK06
JK14A
4K029 AA02 AA04 BA01 BA03 BA06
BA11 BA12 BA17 BA21 BA41
BA54 BA55 BA58 BA64 BB02
BB07 BB08 BD05 CA04 DD06
EA01